Appendix C

Screening Method Validation Study Results

Screening Method Validation Study Results

Lower Fox River Pre-Design Characterization Study

Lower Fox River, Wisconsin

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1 Introduction

This study was conducted as one of the tasks of the RETEC Team in developing the Quality Assurance Project Plan (QAPP) and the Sampling and Analysis Plan (SAP) for the Lower Fox River Pre-design Characterization Study (LFRPD). The LFRPD will involve the sampling of thousands of sediment samples to determine dredge elevations for remedial activities on the Lower Fox River. This study was designed to assess the comparability of the Hybrizymeä Immunoassay (EPA Method 4020) test for PCBs to EPA Method 8082 modified for the Fox River sediment matrix.

The demonstration and utility of Hybrizyme as a screening technology is detailed in the study conducted in August 2000 under the Environmental Technology Verification Program (ETV) by Oak Ridge National Laboratory (ORNL) and the United States Environmental Protection Agency (EPA). The ETV study was performed on soil samples. Sediment samples are often analytically challenging and different from soils, therefore it was important to assess the performance of the Hybrizyme Immunoassay specifically on the Fox River sediment matrix for use in the LFRPD.

The goal was to determine whether the Hybrizyme Immunoassay would yield reliable total PCB results in a specific concentration region. This could then allow it to be used as a means of screening large numbers of sediment samples with only a portion of them requiring full analysis by EPA Method 8082. This would allow for the analysis of a large number of samples in a short period of time at a reduced cost, thus maintaining project schedules and budgets.

In tandem with this study, En Chem also compared the use of the traditional extraction by EPA SW 846 Method 3540C (Soxhlet) and the automated soxhlet extraction by EPA SW 846 Method 3541 using the Soxthermä extraction system (Soxtherm) on Fox River sediments. The extraction method used to date on the Fox River sediment matrix has been the EPA Method 3540C. EPA Method 3541 is automated and provides for a much higher throughput of samples in the lab over the traditional system, and utilizes lesser solvent volume in the extraction process. Again, this helps address maintaining project schedules and budgets. The goal of this portion of the study was to assess the comparability of the Method 8082 (as modified for Fox River sediments) results between sediments prepared by the Soxhlet and Soxtherm extraction methods. Hybrizyme data was also compared to the Soxtherm data.

2 Summary

A variety of conditions were tested and statistical comparisons were made across all concentrations of the study sediments, and specifically around the LFRPD action level of 1.0 mg/kg total PCBs. Soxhlet 8082 modified for the Fox River sediment matrix was used as the "standard" or "true" value for comparison of the Hybrizyme Immunoassay and Soxtherm 8082 results.

Aroclor 1242 is the primary Aroclor found in Lower Fox River sediments, however 1254 and 1260 are also present in some areas of the Lower Fox. As the Hybrizyme test uses a single Aroclor for the calibration curve, Aroclor 1242 was selected for establishment of the calibration curve for this study.

The statistical analysis of the different study conditions was conducted through the SPSS version 11.5 software package. Three different methods were used to evaluate the analytical procedures. The matched pair t-test involved pairing each soxhlet concentration with its matched data generated by Soxtherm, PCB and PCB-XL. The second method involved using regression analysis to determine the correlation coefficient of the line and the line equation for each data pair. The third method was visual examination of the scatter plots generated from the data. Criteria for selection of which screening scenario should be used included: less scatter of data around the decision point, greater correlation by t-test and regression analysis, and more consistency in response to Aroclors.

The data indicate that both Soxtherm and Hybrizyme perform well on Lower Fox River sediments and would provide accurate results in addition to being a cost saving measure for the LFRPD.

3 Methods of Analysis

3.1 Fox River PCB Method-8082 modifications

Fox River sediments have presented unique challenges in sample processing and analysis. This has required the use of air-drying and homogenization steps prior to soxhlet extraction using Method 3540C. Clean up techniques employed on the extracts have included open-column chromatography with Florisil, extraction with elemental copper or shaking with elemental mercury to remove sulfur, and the addition of sulfuric acid to remove contaminants that may interfere with Aroclor identification and quantitation. Analysis is done by gas chromatography with electron capture detection (GC-ECD) using external Aroclor standards. The method detection limit (MDL) achieved by En Chem is 0.022 mg/kg. Whenever Method "8082" is cited in this validation study, it includes these modifications that are also known as the "Fox River PCB Method."

3.2 Hybrizyme Immunoassay

The Hybrizyme PCB Immunoassay kit was selected to conduct tests on Fox River sediments. The original PCB immunoassays used a color development reaction to determine the concentration of PCBs in a sample. The Hybrizyme procedure differs in that it uses the development of fluorescence to determine the PCB concentration. Using the Hybrizyme method with the fluorescence endpoint helps to eliminate possible interferences present, and results in more accurate determinations of PCB concentrations than do immunoassays using the colorimetric endpoint.

The Hybrizyme protocol involves drying a five-gram (dry weight basis) sediment sample by adding sodium sulfate, followed by an extraction with methanol. An aliquot of the sample extract is added to a microtiter plate well and incubated with a PCB antibody. Any PCB present is bound to the PCB antibody. A second antibody attached to the microtiter plate wells binds with and traps the antibody-PCB complex. The microtiter plate wells are washed to remove matrix interferences that may be present in the sample extract. A Europium-labeled PCB compound (PCB Tracer) is added and allowed to bind to any PCB antibody sites that are empty. A second wash step removes any unbound PCB tracer. Enhancement solution is added and forms a highly fluorescent chelate with the europium ions. The amount of fluorescence produced is inversely proportional to the concentration of PCB in the sample. Each extract is analyzed in duplicate. Total PCB concentration is determined by comparing the sample fluorescence to that of a series of Aroclor standards. Hybrizyme uses a Reporting Limit (RL) calculated using the fluorescence readings. The fluorescence readings for all standards and samples are compared to the fluorescence readings for the methanol that was located in wells in the same microtiter well strip. A percent B/B_0 is calculated for each standard or sample. The B/B_0 value equals the fluorescence reading for the

standard or sample ("B") divided by the fluorescence of the methanol ("B_0") times 100. The immunoassay software reports concentrations for samples having percent B/B_0 values between 15% and 90%. Samples with a B/B_0 values greater than 90% are reported as "Low" and samples with B/B_0 values less than 15% are reported as "High." Concentrations reported for samples are based on their B/B_0 percentage and where they fall on the immunoassay calibration curve.

Aroclor 1242 was selected for the standard for establishment of the calibration curve in this method validation study because it is the Aroclor predominantly found in Lower Fox River sediments. The Hybrizyme immunoassay calculates results with the use of either an average calibration curve of stored values that can be regularly updated or a daily calibration curve.

Hybrizyme employs two different protocols for analyzing PCBs, namely PCB and PCB-XL. The PCB protocol was designed for soils and the PCB-XL protocol for tissues. The sensitivity can be adjusted by the selection of the protocol used, and/or varying the amount of sample extract that is used in the immunoassay. The PCB-XL protocol has an Aroclor 1242 Reporting Limit (RL) of 0.05mg/kg on a dry weight basis, however, because of a limited linear range of approximately an order of magnitude, a reduced volume of extract was used in this study. The Aroclor 1242 working range for the PCB-XL protocol, with the reduced extract volume, is approximately 0.4 - 3.5 mg/kg. The PCB protocol calibration working range is approximately 0.4 - 6.0 mg/kg.

3.3 EPA Methods 3540C and 3541

EPA Method 3540C is a soxhlet extraction using methylene chloride as the extraction solvent. The sample is rinse-extracted by the heated solvent circulating through the extractor. The extraction process takes place over a 16 hour period.

EPA Method 3541 is an automated soxhlet extraction method. The Soxtherm extraction system was used in this study. The method uses an automated extraction system to achieve analyte recovery comparable to Method 3540C in a shorter amount of time. The rapid extraction is achieved by the extraction thimble containing the sample being immersed in boiling solvent. The second automated step raises the thimble above the solvent and takes the sample through a rinse-extraction. The final automated step performs the concentration of the extract. The solvent used for the extraction is 4:1 ratio of hexane to acetone.

4 Study Design

4.1 Sample Selection and Randomization

The Scope of the study and the expanded Study Design documents detailing how the study was conducted are in Appendix A. The study was conducted on Lower Fox River sediments collected May 1, 2003 by the RETEC Team specifically for this purpose. It was intended that the study would assess the performance of the Hybrizyme Immunoassay test at action levels of 1.0 mg/kg and 50 mg/kg. The design called for using seven parent samples at each of the action levels aliquoted in triplicate to address various conditions. The sediments collected did not yield samples in the 50 mg/kg concentration region, therefore that concentration region was not studied. A total of parent samples were used in the study. A sample custodian conducted all sample blending, drying, homogenization, aliquoting in triplicate, and random coding. Results were submitted to the En Chem lab manager and the sample numbers were decoded to group and statistically analyze the replicate data. Analysts did not know the identity of the replicates until after all data was generated and submitted.

4.2 Evaluation of Wet vs. Air-Dried Sediments

Samples were selected for the initial portion of the study based on an initial Hybrizyme analysis conducted on wet sediments. Five of the seven parent samples used were generated by blending two discrete samples to reach the target concentration range of 1.0 mg/kg and create sufficient overall sample mass. The blended samples were thoroughly homogenized and split for air-drying. Both the wet and air-dried portions were aliquoted in triplicate, renumbered, and submitted for analysis.

The wet and air dried sediments were analyzed by the PCB and PCB-XL Hybrizyme protocols. Wet and air dried samples were run by the Hybrizyme protocols to determine if there were significant differences in the results, as elimination of the drying step would save significant time and costs. The extraction scenario used was a 3-minute shake followed by 30 minutes in an ultrasonic bath at 30°C. Separate aliquots of the same extract were used to run the analysis by each of the protocols. Sample extracts were quantified against the daily calibration and average calibration scenarios described in the study design. Air-dried sediments were prepared for analysis by Method 8082 using Soxhlet and Soxtherm extraction procedures. The data is summarized in Table 1 and the first set of seven replicate concentrations presented in Table 2.

The evaluation of the Hybrizyme data vs. Method 8082 data after this step showed a higher degree of variability and higher incidence of false negatives at the 1.0 mg/kg action level in the wet sediments as compared to Method 8082. Other issues presented by the wet sediments were keeping the sediment

homogeneous during subsampling, and physical separation of the methanol from the sediment/sodium sulfate mixture because of the amount of sodium sulfate required in the Hybrizyme drying step. Given these results and issues, the subsequent portions of the study were performed on air-dried sediments.

The correlation between the PCB and PCB-XL protocol data was not sufficiently clear at this point to exclude one of the protocols. Data at additional concentration levels was needed. The next step was to analyze additional air-dried sediment replicates to gather data below 1 mg/kg by both Hybrizyme protocols to get a statistical comparison at that level. Both the average calibration and daily calibration protocols were also used to generate results, as more data was necessary to further evaluate the calibration schemes.

4.3 Hybrizyme PCB vs. PCB-XL and Soxhlet vs. Soxtherm

All remaining sediments were air-dried and prepared as the previous samples for submittal for analysis. Analyses were conducted by both Hybrizyme protocols using both the average and daily calibrations for quantitation, Soxhlet 8082, and Soxtherm 8082. The resulting data is presented in Table 2.

Statistical analyses of the data set included the Student t-test on matched pairs and regressions on the same pairs using the statistical package SPSS version 11.5 (Tables 3-22). Scatter plots were also generated to visually assess data correlation (Figures1-8). Each plot shows the best-fit line with the 95% confidence level lines bounding it. Where 8082 data was reported as a non-detect at the MDL of 0.022 mg/kg, one-half of the MDL (0.011 mg/kg) was used as a substitute value for statistical purposes. For Hybrizyme data reported as less than the Reporting Limit (RL), one-half of the RL was used as a substitute value for statistical purposes. Correlation across all values is good for all test cases. However, the primary utility of Hybrizyme to predict 8082 concentrations is in the 0.5 to 2.0 mg/kg range.

Since the concentration range of interest is focused around 1 mg/kg and statistical issues arise when using non-detects, a second set of data analyses was conducted on a subset of the data. The concentration region for the subset was between the MDL for Method 8082 (0.022 mg/kg), and below 3.0 mg/kg.

The study results indicate that the Hybrizyme PCB protocol using a daily calibration and the Soxtherm 8082 data are more closely correlated with Soxhlet 8082 than the other matched pairs (Tables 3-22). Table 28 presents the total PCB and specific Aroclor results for the Hybrizyme PCB and 8082 analyses.

4.4 Ultrasonic Bath Time Study

The Hybrizyme procedure calls for the samples to be shaken in methanol for 3 minutes. The effect on extraction efficiency of following the 3 minute shake by placing the samples in an ultrasonic bath for various lengths of time was assessed for the Hybrizyme PCB protocol. The data from the time study is presented in Table 29.

The four times used were 0, 15, 30 and 45 minutes. The "0 minute" sample extracts were removed from the sample containers after having been shaken vigorously for 3 minutes. Before extracts were removed from the sample containers they were allowed to sit for 5-10 minutes to allow most of the particulates to settle out. At each sampling time, 0.20 ml was removed from each sample container. One sample container was carried through the entire process for each sample. After the fourth sample had been removed, 0.80 ml of the original 25 ml of methanol had been removed from each sample container. No corrections were made in the reported concentrations for this small change (3.2%) in sample volume. The values reported are based on the weight of air-dried sediment used and are not corrected for % solids of the air dried samples.

Some of the samples used for the time study had been diluted in their initial analysis by the Hybrizyme protocols. None of the sample extracts were diluted before being analyzed for the ultrasonic bath time study. It was decided that the extracts shouldn't be diluted for this study because the objective was to determine if the ultrasonic bath would improve the agreement between the Hybrizyme PCB protocol data and that generated by Soxhlet 8082.

The data shows that the ultrasonic bath does not significantly change the concentration of PCB measured by the Hybrizyme immunoassay as compared to the results obtained after the 3 minute shake.

5 Evaluation of Method Comparability

5.1 Statistical Analysis

Various pairings of data were used to statistically assess the comparability of the two different Hybrizyme protocols and the Soxtherm to the Soxhlet prepared samples analyzed by Method 8082. The statistical package SPSS version 11.5 was used to compare the data. Three different methods were used to evaluate the analytical procedures. The matched pair t-test involved pairing each Soxhlet concentration with its matched data generated by Soxtherm, PCB and PCB-XL. The second method involved using regression analysis to determine the correlation coefficient of the line and the line equation for each data pair. The third method was visual examination of scatter plots generated from the matched pair data. Criteria for selection of which screening method should be used included: less scatter of data around the decision point, greater correlation by t-test and regression analysis, and more consistency in response to Aroclors.

Correlation across all values is good when all test cases are included. However, when focusing on concentrations above the Method 8082 MDL but below 3 mg/kg, the following observations were made:

Statistical analyses of the full data set included Student t-test on matched pairs and regressions on the same pairs using the statistical package SPSS version 11.5 (Tables 3-12). Where method 8082 data was reported as a non-detect at the MDL, one-half of the MDL (0.011 mg/kg) was used as a substitute value for statistical purposes. For Hybrizyme data reported as less than the RL, one-half of the RL was used as a substitute value for statistical purposes. Since the concentration range of interest is focused around 1 mg/kg and statistical problems occur when using non-detect values, a second set of data analyses was conducted on a subset of the data. The concentration region used for the subset was from the MDL for Soxhlet 8082 to 3.0 mg/kg (Tables 13-22).

Scatter plots were also generated to assess data correlation (Figures 1-8). Each plot shows the best fit line with the 95% confidence level lines bounding it. The plots that are focused on the 0.011 – 3.0 mg/kg concentration range show a higher correlation around the 1.0 mg/kg action level than at other concentration points.

The means of the triplicate values were also analyzed as this data set will have less statistical noise. The mean data set for total PCBs by Soxhlet and Soxtherm 8082 and both Hybrizyme protocols are presented in Table 30, with results of the t-test and regression analysis in Tables 23-26. These data further support the use of the Hybrizyme PCB protocol with the daily calibration and show that it correlates well to both the Soxhlet 8082 and Soxtherm 8082 data.

Analysis of the data from the wet sediments is presented in Table 27. The wet sediment data does not correlate as well with Soxhlet 8082 as the air-dried sediments. The wet sediments also posed handing problems, these factors eliminated further testing of the wet sediments for comparability.

5.2 False Positive/False Negative Results

False positive and false negative results were assessed for all protocols on air-dried samples at both the action level of 1.0 mg/kg and at the Soxhlet 8082 MDL of 0.022 mg/kg. The results are summarized below:

Action Level = 1.0 mg/kg – Total Data Set

	Soxhlet 8082	Soxtherm 8082	PCB Ave Cal.	PCB Daily Cal.	PCB-XL Ave Cal.	PCB-XL Daily Cal.
False Positives (n=57)	0	0	1	1	1	1
False Negatives (n=57)	0	1	6	4	3	4
True (n=57)	57	56	50	52	53	52

Soxhlet was assumed to be the "true" value. All results are based upon the 1.0 mg/kg decision level.

Action Level 1.0 mg/kg – Data Set >0.011 mg/kg and <3.0 mg/kg

	Soxhlet 8082	Soxtherm 8082	PCB Ave Cal.	PCB Daily Cal.
False Positives (n=25)	0	0	1	1
False Negatives (n=25)	0	1	4	5
True (n=25)	25	24	20	19

Soxhlet was assumed to be the "true" value. All results are based upon the 1.0 mg/kg decision level.

Soxhlet 8082 MDL 0.022 mg/kg – Total Data Set

	Soxhlet 8082	Soxtherm 8082	PCB Ave Cal.	PCB Daily Cal.	PCB-XL Ave Cal.	PCB-XL Daily Cal.
False Positives (n=57)	0	1	7	6	6	6
Largest Difference (mg/kg)	0	.05	.6	.5	.72	.71
False Negatives (n=57)	0	1	4	5	5	4
Largest Difference (mg/kg)	0	.03	.08	.05	.20	.20
True (n=57)	57	55	46	46	46	47

Soxhlet was assumed to be the "true" value. All negative results are based upon the Soxhlet 8082 MDL of 0.022 mg/kg. One half the MDL (0.011 mg/kg) was entered in the database for 8082 non-detects. The Soxtherm 8082 MDL was assumed to be the same as the Soxhlet 8082 MDL. The Hybrizyme RL ranges from 0.38-0.50 mg/kg. The largest difference on false positive is the largest positive concentration on the comparison test when Soxhlet 8082 was <MDL. Largest difference on the false negative is the largest concentration on the Soxhlet when the comparison test was negative.

6 Quality Assurance/Quality Control

Table 31 summarizes the Method Blanks (MB), Laboratory Control Spikes (LCS), and sample duplicates run during the study. Table 32 summarizes the Matrix Spikes (MS) and Matrix Spike Duplicates (MSD). The MB is performed by taking Ottawa Sand through the sample preparation and analysis steps. LCS's were prepared by spiking Aroclor 1242 into Ottawa sand and taken through the analysis as a sample. All LCS's and MS/MSD's were spiked at the 1.0 mg/kg action level of the LFRPD with Aroclor 1242. All QA/QC checks throughout the study yielded very good results as is evidenced in the tables. The QA/QC checks did not give reason to throw out any of the data generated in the study. However, a larger data set would be needed to calculate statistically defensible acceptance limits. The proposed initial frequency for the Hybrizyme PCB protocol batch QC is 1 for every 20 samples for each of the following checks with the associated proposed initial acceptance limits:

Calibration Check at 200ug/l	80-120 % recovery
Method Blank	< RL (0.5 mg/kg)
LCS at 1.0 mg/kg 1242	70-130 % recovery
Lab duplicate	<30% RPD
MS/MSD at 1.0 mg/kg 1242	60-120 % recovery

Additionally it is suggested that if Hybrizyme is used for the LFRPD, a Fox River control sediment sample be analyzed daily to continually assess the performance of the method.

7 Aroclor Response Comparison

A comparison of Aroclor 1242, 1254, and 1260 response was conducted using the Hybrizyme PCB and PCB-XL protocols. Aroclor 1242, 1254 and 1260 standards were prepared in methanol. The standards were analyzed using both the Hybrizyme PCB and PCB-XL protocols. Daily calibration curves were generated for both protocols using the Aroclor 1242 analytical data. These calibration curves were used to calculate concentrations of the Aroclor 1254 and 1260 standards. Results of the analysis are provided in Table 33.

Smaller differences between the actual standard concentration and analyzed concentration were found for the Aroclor 1254 and 1260 standards when using the Hybrizyme PCB protocol. Aroclor 1254 gave an average response 2.9 times greater than Aroclor 1242 and Aroclor 1260 yielded an average response 1.7 times greater. When the same standards were analyzed using the Hybrizyme PCB-XL protocol, Aroclor 1254 gave an average response 5.7 times the Aroclor 1242 response and Aroclor 1260 resulted in an average response that was 6.4 times greater. Hybrizyme indicates in their "Instructions and User Guide" for the PCB protocol that Aroclor 1254 has a response 4.0 times that of Aroclor 1242 and Aroclor 1260's response is 3.3 times that of Aroclor 1242. The differences in response between the Aroclors using the PCB analysis protocol found in this study were smaller than those reported by Hybrizyme. This may be due to small changes in the analysis reagents since the original Hybrizyme method performance data was generated.

Hybrizyme has indicated that the PCB-XL protocol was developed for use in analyzing PCBs in biological tissue samples. In designing the test, they developed a system that would give a greater response to the more highly chlorinated PCB congeners. This study's results are in agreement with this. Based on the greater similarity of response for the various Aroclors when analyzed by the Hybrizyme PCB protocol, it was selected over the PCB-XL protocol for use in the final validation step of the study. The purpose of the final validation using the selected method (Hybrizyme PCB with daily calibration) was to assess its reproducibility and recommend the frequency of analysis by Method 8082 at various concentration regions to confirm the screening method result.

7.1 Confirmation Rate

Regression analysis was done on a variety of data pairing scenarios (Tables 3-22) to assess the mathematical relationship between Hybrizyme and Soxtherm 8082 to Soxhlet 8082. Correlation across all values is good for all tests. To determine a confirmation rate for the Hybrizyme PCB protocol, the data assessment was focused on results above the 8082 MDL and less than 3.0 mg/kg. Hybrizyme replicate analyses were performed at various points during the study to ensure data reproducibility. As a final step in the study, a set of

samples were reanalyzed using the chosen protocol of: air-dried sediments, Hybrizyme PCB method, and a Daily Calibration curve. The regression analysis comparing Soxhlet 8082 to Hybrizyme PCB protocol/Daily Calibration in the 0.011 – 3.0 mg/kg concentration range was used to generate expected Soxhlet 8082 data with the Hybrizyme PCB result as the predictor. This was done for Hybrizyme concentrations from 0.2 mg/kg to 2.5 mg/kg. The resulting expected range of Soxhlet 8082 concentrations is presented in Table 34.

Aroclor 1242 is the predominant Aroclor present in the overall sample set used in the study as well as the final validation sample set. Total PCB concentrations ranged from the 8082 MDL to 3.0 mg/kg. The recommendation for the initial criteria for determining whether samples analyzed by Hybrizyme require confirmatory analysis by Method 8082 is:

- 100 % of samples > 0.5 to < 2.0 mg/kg
- 5 % of samples $\leq$ 0.5 mg/kg
- 5 % of samples $\geq$ 2.0 mg/kg.

If confirmation samples and/or the control sediment recovery indicate that the Hybrizyme PCB screening method performance is drifting outside of the ranges determined by this study, then the percentage of samples taken for confirmation would be increased on the false negative or false positive side, in correspondence with the risk of error seen.

The study data was assessed to recommend confirmation criteria when Aroclor 1254 and/or Aroclor1260 are present based on the Aroclor response comparison. A subset of the study samples contained Aroclor 1254, none contained Aroclor 1260. In all samples where both Aroclors 1242 and 1254 were found, Aroclor1242 was always at a predominantly higher concentration (4x to 8x) than Aroclor1254. In each of these instances the above criteria for confirmation hold true. There would not have been a false positive for total PCBs at the 1.0 mg/kg action level due to the presence of Aroclor 1254.

To further assess samples with multiple Aroclors present, the Wisconsin Tissue Mills (WTM) 2000-2001 data set (data submitted to WDNR by WTM as comment to the Proposed Remedial Action Plan, October 2001) was used. It should be noted that the data in this set were also generated by En Chem using Method 8082 modified for Fox River sediments. From this data set of 346 samples by Aroclor, a selection was made of samples falling into the >0.3 to <6.0 mg/kg total PCB range. The MDL of this data set is 0.022mg/kg. This selected range, as the samples used in this study, targets the low concentration region of interest. The resulting subset contains 20 samples. The percent composition by Aroclor of the WTM subset shows that Aroclor 1242 is the most predominant, this was also the case in the study data for the low concentration region of concern.

The next step in the analysis of the WTM data subset was to use the mathematical relationship between Method 8082 and Hybrizyme that was developed with the study samples (see Section 7.2). This was done to mathematically predict the associated Hybrizyme total PCB concentration based on the WTM total 8082 PCB concentration. This showed that Hybrizyme would correctly identify the WTM subset samples for Method 8082 confirmatory analysis, based on the study recommended range of >0.5 to <2.0 mg/kg, with no false positives or negatives at the 1.0 mg/kg action level. The summary of this data is presented in Table 35. Figure 14 presents a graphical representation of this data bounded by the 99% Confidence Interval.

7.2 Reproducibility and Refinement of Confirmation Rate

Various samples were re-extracted and re-analyzed during the course of the study to determine if it was possible to reproduce total PCB concentrations produced by Hybrizyme. A set of 21 samples were analyzed at the end of the study using the protocol that has given the best correlation to Soxhlet 8082 (Hybrizyme PCB Protocol, 3-minute shake, no ultrasonic bath, daily calibration curve) for a final assessment of reproducibility. All sample concentrations > Soxhlet 8082 MDL and < 3.0 mg/kg, including all duplicate analyses on the PCB Hybrizyme test, were included along with the initial data validation data. A final regression analysis was generated using all of this data in order to determine the best-fit equation with the 95 and 99% confidence intervals. The replicates indicate that the Hybrizyme PCB protocol is highly reproducible Table 34.

Figure 13 graphically displays the mean regression line with the 95 and 99% confidence intervals. Reference lines at 1.0 mg/kg for Method 8082 and 2.0 mg/kg for the PCB Hybrizyme methods indicate where the intersection of these two values occurs relative to the acceptance window. The 2.0 mg/kg concentration as a decision point is clearly a conservative number for the initial screen. This analysis confirms the initial recommendation of all samples in the concentration range of >0.5 to <2.0 mg/kg total PCB by the Hybrizyme PCB protocol be confirmed by Soxhlet or Soxtherm 8082.

This regression analysis resulted in a line equation between the PCB Hybrizyme-Daily Cal vs. Soxhlet of: **Soxhlet=1.414*(PCB Hybrizyme Daily Cal)-0.335**. The correlation coefficient for this line is 0.689. Two outliers were identified during this analysis, both on Replicate No. 57. This sample had a high screen result, outside the acceptance window of > 2.0 mg/kg. In order to conservatively set the range where samples would be confirmed by Method 8082 the outliers were not excluded from the data set.

As more precision and accuracy data are generated, they can be used to refine the decision points at which samples should be confirmed via Method 8082. Most of the confirmation samples should be selected from the range of concentration between the 95 to 99% confidence limits to ensure minimal false positives and false negatives at the 1.0 mg/kg action level.

8 Sample Throughput

The various analysis schemes were assessed for sample throughput in the laboratory. The Soxhlet extraction is the most production limiting step in the analysis because of the length of the extraction time. The throughput is based on having sufficient air-dried samples to run full (n = 20 field samples and QC) sample sets. Based on the anticipated sample collection rate for the LFRPD, sufficient air-dried/homogenized samples for ongoing full samples sets should be available to the laboratory within the first week of sampling. Throughput estimates are best case scenarios based on one 8-hour shift, 5 days/week. Adding a second shift would double the throughput estimates for Hybrizyme and Soxtherm, but does not increase the throughput for Soxhlet. Throughputs are for preparation and analysis of samples, but do not include final data package preparation and internal QC review:

Method	Throughput	Estimated Cost/Sample
Soxhlet extraction	200 samples/week	$125
Hybrizyme PCB Protocol	60 samples/day	$40
Soxtherm extraction	60 samples/day	$105

9 References

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Appendix A

Preliminary Scope for Hybrizyme PCB Screen Method Development Study

Goal

Assess Hybrizyme PCB Screen Method to determine comparability to the Fox River PCB Method to provide reliable results. This would allow its use to screen large numbers of samples at the determined action levels, ultimately resulting in significant cost savings and keeping the project on schedule.

Parameters to assess in the Study include:

- Wet vs. air-dried sediments;
- Hybrizyme PCB vs. PCB-XL Protocol;
- Length of time in ultrasonic bath;
- Aroclor mixtures including Aroclor 1242, 1254, and 1260;
- QA/QC requirements; and
- Sample throughput utilizing Hybrizyme in conjunction with the Fox River PCB Method.

Study Design

I. Obtain native Fox River sediments, 7 at each action level:

-1 ppm action level
-50 ppm action level

II. Split each native sediment sample into two portions:

-Wet sediment; and
-Air dried sediment.

III. Aliquot each portion in triplicate, randomly code and submit for analysis:

-Hybrizyme PCB Protocol; and
-Hybrizyme PCB-XL Protocol.

IV. Length of sonication for each Protocol:

-Assess ultrasonic bath time.

V. Calibration criteria to assess the data against:

-Average calibration using Aroclor 1242;

-Daily calibration using Aroclor 1242;

-Daily 1 ppm or 50 ppm standard;

-RL for curve centered around 1ppm action level based on low standard at 0.5ppm; and

-RL for curves of Aroclor mixture 1242, 1254, and 1260.

VI. QC Checks:

-Laboratory Control Sample (1 in 20) run at the action level (1ppm or 50 ppm);

-Sample Duplicates (1 in 20);

-Initial acceptance criteria 70-130% until sufficient data is produced to calculate limits;

-Laboratory Control Blank using Ottawa Sand (1 in 20); and

-MS/MSD - clean sediment spiked at action level to set error bars on the test.

VII. Statistical Assessment of Data via SPSS software Version 11.5:

-All screen conditions will be tested for statistical equivalence to the Fox River PCB Method; and

-Parameters assessed will be the variance of each method, range of data scatter and false positive/false negative values.

VIII. Establish criteria for Hybrizyme data acceptance and confirmation rate by Fox River PCB Method:

-Screen method RL= 0.5 ppm (based on low standard), reanalyze X% of samples in the 0.5-1.5 ppm range by Fox River PCB Method;

-5% of samples with results <0.5 ppm; and
-5% of samples with results >1.5ppm.

If confirmation samples show that test performance is drifting outside of ranges determined by the initial studies, then the percentage of samples taken for confirmation would be increased on the false negative or false positive side, in correspondence with the risk of error seen.

IX. Study Report summarizing findings including cost and laboratory throughput.

Study Design

The following information expands on how the first four points of the "Preliminary Scope for Hybrizyme PCB Screen Method Modification Study" Appendix C of the Scope of Work submitted by The RETEC Team will be performed. It also includes comments on how samples that contain significant amounts of Aroclors 1254 and 1260 will be handled. This responds to the last point in Roman Numeral V of the design.

The analysis of all samples for parts 1, 2 and 3 of the Study Design will be done by the initial study condition of extraction of the PCBs from the samples using methanol. The samples will be shaken vigorously for three minutes followed by a thirty minute sonication in an ultrasonic bath (Branson Model 8210).

Obtaining Samples in the Proper Concentration Ranges

1.) Native samples are obtained. A portion of each is air-dried and homogenized. The samples are analyzed by the Hybrizyme immunoassay (SW846 Method 4020) PCB Protocol to obtain information on whether the samples are in the concentration ranges desired. The goal is to have seven sediments selected that are in the 0.5-3 ppm range and another seven that are in the 30-80 ppm range. Based on the information obtained, the samples that are acceptable will be homogenized, portions air-dried, aliquots separated and numbered. If appropriate concentration sediments were not obtained, blending will be employed to produce the desired concentrations.

Air-dried and wet sediment samples will be analyzed by the Hybrizyme immunoassay protocols (PCB and PCB-XL). Air-dried sediments only will be analyzed by the Fox River PCB Method (SW846 Method 8082).

Analysis of Wet vs. Air-Dried Sediments

2). Air-dried sediments will be analyzed by the two Hybrizyme PCB immunoassay protocols and the Fox River PCB Method. Three separate aliquots of each air-dried portion of the native samples will be prepared and analyzed by the initial study condition.

3.) Wet sediments will be analyzed by the two Hybrizyme PCB immunoassay protocols. Three separate aliquots of each wet portion of the native samples will be prepared and analyzed by the initial study condition.

Determination of Best Hybrizyme Method and Wet vs. Air-Dried

4.) Results of these two studies will have statistical analyses conducted to determine if there is significant difference between the results obtained from the PCB and PCB-XL immunoassay protocols. The air-dried and wet sediment results will also be compared to determine if they are statistically the same or different at the 95% confidence level using the Student t-test. Based on the results of the statistics and comparison with the Fox River Method data, decisions will be made by the RETEC Team on which immunoassay protocol

will be used for the additional portions of the study. It will also be decided whether the additional portions of the study will be conducted on air-dried or wet sediments.

If it is determined that the PCB and PCB-XL immunoassay protocols provide results that are not statistically different, the Hybrizyme PCB protocol will be used for the remainder of the study. This method requires less than half the time to conduct as the PCB-XL protocol and does not require dilution of samples to obtain results for the 1 ppm action limit samples. Regarding the air-dried and wet sediments, if they show no significant statistical difference at the 95% confidence level, the additional portions of the study will be conducted on wet sediments. Analyzing wet sediments would eliminate the need to air dry sediments before conducting PCB immunoassay analyses. Those sediments that would undergo analysis by the Fox River Method would require air-drying before being analyzed. If the results of the comparisons of these two sets of data show statistical differences, the protocol and sample type that yield the best comparison with results obtained by the Fox River Method will be employed during the remainder of the study. All samples will be quantified using three different standardization routines, 1) daily calibration; 2) stored average calibration; 3) daily check standard at the action level of 1 ppm or 50 ppm. All results will be statistically compared to the Fox River Method to see which quantitation protocol delivers the best correspondence.

Extraction Optimization

5.) Length of sonication by ultrasonic bath will be studied. Data from an earlier study was generated using a 30-minute sonication in a Branson 8210 ultrasonic cleaner. Suggested additional ultrasonic bath times to be tried are 0, 15 and 45 minutes.

Data obtained at the various times will be statistically analyzed to determine if the data sets are statistically the same at the 95% confidence level using the Student T test. The sonication time that provides the best comparison with the data obtained from the Fox River Method will be selected as the time that would be used in the analysis of samples from OU1.

Verification of Aroclor 1254 and 1260 Responses

6.) Information is available from Hybrizyme on the variation in response of the various Aroclors when tested using the Hybrizyme PCB immunoassay protocol. Response values that they have determined for the various Aroclors follows:

Aroclor	% Reactivity
1016	25
1221	10
1232	20
1242	40
1248	100
1254	160
1260	130
1261	110

Aroclor 1254 and 1260 standards will be prepared. They will be analyzed using the Hybrizyme protocol chosen based on the statistical analysis conducted in Item 4 above. Quantitation for Aroclor 1254 and 1260 will be based on a calibration curve generated using Aroclor 1242 standards. The results will be compared to the % Reactivity data that has been generated by Hybrizyme. Results of this study will be used to establish a different concentration range to be used to determine when confirmation analysis of samples by the Fox River Method will be employed for samples expected to contain a significant amount of Aroclors 1254 and/or 1260. Assuming the study generates data that agrees with the data supplied by Hybrizyme (+/-25% at the ED50 value), the following concentration range is proposed:

- Screen method RL = 0.5 ppm (based on Aroclor 1242 low standard)
- Analyze X percent of samples in the 0.5 to 3.0 ppm range by the Fox River Method (SW846 Method 8082). This concentration range is based on analyzing the samples using an Aroclor 1242 calibration curve.
- Analyze 5 percent of samples with results less than 0.5 ppm
- Analyze 5 percent of samples with results greater than 3.0 ppm

The reason for the change in the concentration range at which sediments containing significant amounts of Aroclors 1254 and/or 1260 will be analyzed by the Fox River Method is the differing response of the PCB immunoassay to the various Aroclors. Because there are known areas in OU1 that contain significant amounts of Aroclor 1254 and/or 1260, the concentration range at which sediments from those areas will be confirmed by the Fox River Method has been widened (0.5 – 3.0 ppm). This is done to avoid removing more sediment for those regions of OU1 than necessary.

Determination and Refinement of Confirmation Rate

7.) The confirmation rate of immunoassay data by the Fox River PCB Method for the 1 ppm action level is proposed in Roman Numeral VIII of the Study outline. Similar guidelines for the 50 ppm action level will need to be established. At this time there is not immunoassay data for samples in that concentration range. Guidelines utilizing the data generated in the study will be provided.

After the study is completed and the final protocol is accepted, the results generated will be used to set 95 and 99 % confidence levels of the mean compared to the Fox River Method. A final validation protocol using the Hybrizyme method of choice will be performed. This validation will be employ three (3) aliquots each of the seven (7) native samples around the two (2) action limits . If the results are equivalent statistically to the initial data obtained, the data will be pooled and new 95 and 99% confidence limits will be set. With each batch of samples analyzed, at least one of these study/control sediments should be analyzed per batch and the results used to update the statistical pool. As limits change with more data points, these can be used to set decision points for which samples should be confirmed via the Fox River Method. Confirmation samples should be highest in the range of concentrations between the 95 to 99% confidence limits to ensure minimal false positives and false negatives.

Table 1
Total PCB Data for Wet Sediments by Hybrizyme Protocol and Calibration Routine

			Wet Sediment-PCB		Wet Sediment-PCB-XL		Air-dried Sediment Total PCB	
Sample No.	Replicate No.	% Solids	Daily Cal. (mg/kg)	Ave. Cal. (mg/kg)	Daily Cal. (mg/kg)	Ave. Cal. (mg/kg)	Soxhlet 8082 (mg/kg)	Soxtherm 8082 (mg/kg)
1	1	22.6	1.01	1.07	0.69	0.67	2.1	2.3
1	10	22.5	0.88	0.93	0.66	0.64	1.8	1.8
1	15	22.5	0.83	0.87	0.78	0.75	2.0	1.7
2	2	31.1	5.36	5.94	5.01	4.53	12	16
2	8	30.8	4.59	5.06	5.34	4.80	12	14
2	21	30.8	4.51	4.97	4.59	4.17	12	16
3	3	31.3	5.20	5.76	5.10	4.62	12	16
3	13	31.9	5.32	5.89	5.52	4.95	12	16
3	17	31.8	5.09	5.63	5.82	5.22	12	16
4	4	26.1	1.10	1.16	0.83	0.79	3.3	3.2
4	11	25.9	0.86	0.90	0.88	0.84	3.2	3.3
4	20	26.2	0.91	0.95	0.91	0.86	3.4	2.6
5	5	23.7	0.74	0.77	0.61	0.59	1.5	1.2
5	12	23.3	0.72	0.75	0.75	0.72	1.1	1.2
5	16	23.6	0.72	0.75	0.62	0.61	1.2	1.1
6	6	24.6	0.59	0.61	0.68	0.65	1.1	0.99
6	14	24.6	0.60	0.62	0.57	0.56	1.1	1.1
6	19	24.6	0.52	0.54	0.58	0.57	1.1	1.0
7	7	28.9	1.08	1.14	1.46	1.33	2.6	2.3
7	9	28.8	1.10	1.16	1.22	1.13	2.0	2.5
7	18	28.8	1.04	1.10	1.56	1.42	2.9	2.6
	MB		<0.45	<0.46	<0.37	<0.38		
	LCS (1.0 mg/kg)		0.96(96%)	1.01(101%)	0.94(94%)	0.89(89%)		

All values reported on a dry weight basis.

Table 2
Total PCB Data of Individual Air-Dried Replicates by 8082 and Hybrizyme Protocols by Calibration Routine

Sample No.	Replicate No.	Soxhlet 8082 Total PCB (mg/kg)	Soxtherm 8082 Total PCB (mg/kg)	PCB Ave Cal Total PCB (mg/kg)	PCB Daily Cal Total PCB (mg/kg)	XL Ave Cal Total PCB (mg/kg)	XL Daily Cal Total PCB (mg/kg)
1	1	2.1	2.3	1.11	1.27	1.5	1.37
1	10	1.8	1.8	1.14	1.29	1.12	1.07
1	15	2	1.7	1.09	1.24	1.08	1.03
2	2	12	16	6.09	6.69	8.01	6.72
2	8	12	14	6.46	7.09	8.91	7.33
2	21	12	16	6.15	6.78	6.54	5.65
3	3	12	16	6.09	6.78	9.45	7.79
3	13	12	16	7.04	7.76	9.36	7.73
3	17	12	16	7.26	7.98	7.62	6.5
4	4	3.3	3.2	1.23	1.4	1.14	1.08
4	11	3.2	3.3	1.19	1.35	1.41	1.3
4	20	3.4	2.6	1.11	1.27	1.26	1.18
5	5	1.5	1.2	0.86	0.98	0.92	0.9
5	12	1.1	1.2	0.95	1.08	1.45	1.33
5	16	1.2	1.1	0.84	0.96	1.09	1.04
6	6	1.1	0.99	0.88	1.01	1.02	0.99
6	14	1.1	1.1	0.8	0.92	0.79	0.77
6	19	1.1	1	0.75	0.87	0.88	0.87
7	7	2.6	2.3	1.33	1.5	2.01	1.75
7	9	2	2.5	1.21	1.37	1.27	1.18
7	18	2.9	2.6	1.28	1.44	2.27	1.94
8	22	0.011	0.01	0.21	0.19	0.72	0.83
8	39	0.011	0.011	0.21	0.18	0.22	0.2
8	54	0.011	0.011	0.5	0.49	0.22	0.19
9	23	18	22.8	22	20	24	22
9	45	17.9	21.7	19	17	26	23
9	49	15.7	23.7	17	17	19	18
10	24	0.011	0.011	0.42	0.385	0.22	0.34
10	38	0.011	0.048	0.36	0.33	0.34	0.3
10	51	0.025	0.011	0.21	0.2	0.22	0.19
11	25	0.039	0.066	0.21	0.19	0.22	0.5
11	44	0.075	0.11	0.21	0.18	0.22	0.2

Table 2
Total PCB Data of Individual Air-Dried Replicates by 8082 and Hybrizyme Protocols by Calibration Routine

Sample No.	Replicate No.	Soxhlet 8082 Total PCB (mg/kg)	Soxtherm 8082 Total PCB (mg/kg)	PCB Ave Cal Total PCB (mg/kg)	PCB Daily Cal Total PCB (mg/kg)	XL Ave Cal Total PCB (mg/kg)	XL Daily Cal Total PCB (mg/kg)
11	52	0.054	0.068	0.21	0.2	0.54	0.48
12	26	0.16	0.16	0.45	0.42	0.63	0.73
12	37	0.095	0.11	0.59	0.53	0.45	0.39
12	50	0.134	0.09	0.55	0.54	0.22	0.19
13	27	8	10.1	5	4.9	5.9	6.2
13	43	7.4	9.3	5.5	5.4	8.4	7.8
13	46	6.29	9.8	3.9	3.7	7.1	6.5
14	28	0.011	0.011	0.21	0.19	0.22	0.43
14	36	0.011	0.011	0.54	0.48	0.22	0.2
14	47	0.011	0.011	0.21	0.18	0.46	0.41
15	29	5.75	7.1	2.5	2.7	4.6	4.7
15	42	4.83	5.02	3.8	3.7	5.3	4.9
15	48	3.51	5.99	3.6	3.5	5.6	5.1
16	30	0.224	0.28	0.51	0.48	0.76	0.87
16	35	0.16	0.24	0.5	0.45	0.52	0.46
16	57	0.28	0.26	1.57	1.53	4.1	4.1
17	31	0.011	0.011	0.21	0.19	0.6	0.71
17	41	0.011	0.011	0.21	0.18	0.22	0.2
17	56	0.011	0.011	0.6	0.59	0.47	0.41
18	32	0.27	0.13	0.52	0.49	0.73	0.84
18	34	0.2	0.323	0.46	0.41	0.22	0.2
18	53	0.213	0.22	0.65	0.64	0.64	0.57
19	33	0.011	0.011	0.45	0.4	0.22	0.2
19	40	0.011	0.011	0.21	0.18	0.22	0.2
19	55	0.011	0.011	0.31	0.21	0.22	0.21

All data reported on a dry weight basis.

Table 3 T-Test-Match Pairs on all Dry Weight Data

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	Soxhlet 8082	3.3660	57	5.01522	.66428
	Soxtherm 8082	4.2221	57	6.64631	.88032
Pair 2	Soxhlet 8082	3.3660	57	5.01522	.66428
	PCB Ave Cal	2.604	57	4.4946	.5953
Pair 3	Soxhlet 8082	3.3660	57	5.01522	.66428
	PCB Daily Cal	2.6204	57	4.27959	.56685
Pair 4	Soxhlet 8082	3.3660	57	5.01522	.66428
	XL Ave Cal	3.3165	57	5.49718	.72812
Pair 5	Soxhlet 8082	3.3660	57	5.01522	.66428
	XL Daily Cal	3.0223	57	4.94317	.65474
Pair 6	Soxtherm 8082	4.2221	57	6.64631	.88032
	PCB Ave Cal	2.604	57	4.4946	.5953
Pair 7	Soxtherm 8082	4.2221	57	6.64631	.88032
	PCB Daily Cal	2.6204	57	4.27959	.56685
Pair 8	Soxtherm 8082	4.2221	57	6.64631	.88032
	XL Ave Cal	3.3165	57	5.49718	.72812
Pair 9	Soxtherm 8082	4.2221	57	6.64631	.88032
	XL Daily Cal	3.0223	57	4.94317	.65474

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	Soxhlet 8082 & Soxtherm 8082	57	.993	.000
Pair 2	Soxhlet 8082 & PCB Ave Cal	57	.917	.000
Pair 3	Soxhlet 8082 & PCB Daily Cal	57	.943	.000
Pair 4	Soxhlet 8082 & XL Ave Cal	57	.924	.000
Pair 5	Soxhlet 8082 & XL Daily Cal	57	.913	.000
Pair 6	Soxtherm 8082 & PCB Ave Cal	57	.917	.000
Pair 7	Soxtherm 8082 & PCB Daily Cal	57	.945	.000
Pair 8	Soxtherm 8082 & XL Ave Cal	57	.922	.000
Pair 9	Soxtherm 8082 & XL Daily Cal	57	.912	.000

Table 3 T-Test-Match Pairs on all Dry Weight Data (Continued)

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	Soxhlet 8082 - Soxtherm 8082	-.8561	1.75998	.23311	-1.3230	-.3891	-3.672	56	.001
Pair 2	Soxhlet 8082 - PCB Ave Cal	.7616	1.99835	.26469	.2314	1.2919	2.877	56	.006
Pair 3	Soxhlet 8082 - PCB Daily Cal	.7456	1.73117	.22930	.2862	1.2049	3.252	56	.002
Pair 4	Soxhlet 8082 - XL Ave Cal	.0495	2.09652	.27769	-.5068	.6058	.178	56	.859
Pair 5	Soxhlet 8082 - XL Daily Cal	.3437	2.07349	.27464	-.2064	.8939	1.252	56	.216
Pair 6	Soxtherm 8082 - PCB Ave Cal	1.6177	3.09455	.40988	.7966	2.4388	3.947	56	.000
Pair 7	Soxtherm 8082 - PCB Daily Cal	1.6016	2.95713	.39168	.8170	2.3863	4.089	56	.000
Pair 8	Soxtherm 8082 - XL Ave Cal	.9056	2.65588	.35178	.2009	1.6103	2.574	56	.013
Pair 9	Soxtherm 8082 - XL Daily Cal	1.1998	2.94354	.38988	.4188	1.9808	3.077	56	.003

Table 4 Regression-Soxhlet vs. Soxtherm on Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	Soxtherm 8082(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.993(a)	.987	.987	.57855

a Predictors: (Constant), Soxtherm 8082

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1390.129	1	1390.129	4153.172	.000(a)
	Residual	18.409	55	.335		
	Total	1408.539	56			

a Predictors: (Constant), Soxtherm 8082
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.201	.091		2.208	.031
	Soxtherm 8082	.750	.012	.993	64.445	.000

a Dependent Variable: Soxhlet 8082

Table 5 Regression-Soxhlet vs. PCB Daily Calibration on Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.943(a)	.889	.887	1.68714

a Predictors: (Constant), PCB Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1251.985	1	1251.985	439.842	.000(a)
	Residual	156.554	55	2.846		
	Total	1408.539	56			

a Predictors: (Constant), PCB Daily Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.471	.263		1.792	.079
	PCB Daily Cal	1.105	.053	.943	20.972	.000

a Dependent Variable: Soxhlet 8082

Table 6 Regression-Soxtherm vs. PCB Daily Calibration on Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.945(a)	.893	.891	2.19842

a Predictors: (Constant), PCB Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	2207.892	1	2207.892	456.833	.000(a)
	Residual	265.817	55	4.833		
	Total	2473.710	56			

a Predictors: (Constant), PCB Daily Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.377	.342		1.102	.275
	PCB Daily Cal	1.467	.069	.945	21.374	.000

a Dependent Variable: Soxtherm 8082

Table 7 Regression-Soxhlet vs. PCB Average Calibration on Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.917(a)	.842	.839	2.01357

a Predictors: (Constant), PCB Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1185.544	1	1185.544	292.405	.000(a)
	Residual	222.995	55	4.054		
	Total	1408.539	56			

a Predictors: (Constant), PCB Ave Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.700	.309		2.266	.027
	PCB Ave Cal	1.024	.060	.917	17.100	.000

a Dependent Variable: Soxhlet 8082

Table 8 Regression-Soxtherm vs. PCB Average Calibration on Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.917(a)	.841	.838	2.67193

a Predictors: (Constant), PCB Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	2081.054	1	2081.054	291.497	.000(a)
	Residual	392.656	55	7.139		
	Total	2473.710	56			

a Predictors: (Constant), PCB Ave Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.690	.410		1.683	.098
	PCB Ave Cal	1.356	.079	.917	17.073	.000

a Dependent Variable: Soxtherm 8082

Table 9 Regression-Soxhlet vs. PCB-XL Average Calibration, Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.924(a)	.855	.852	1.92904

a Predictors: (Constant), XL Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1203.874	1	1203.874	323.519	.000(a)
	Residual	204.665	55	3.721		
	Total	1408.539	56			

a Predictors: (Constant), XL Ave Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.569	.299		1.901	.062
	XL Ave Cal	.843	.047	.924	17.987	.000

a Dependent Variable: Soxhlet 8082

Table 10 Regression-Soxtherm vs. PCB-XL Average Calibration, Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.922(a)	.849	.846	2.60400

a Predictors: (Constant), XL Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	2100.764	1	2100.764	309.809	.000(a)
	Residual	372.946	55	6.781		
	Total	2473.710	56			

a Predictors: (Constant), XL Ave Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.527	.404		1.305	.197
	XL Ave Cal	1.114	.063	.922	17.601	.000

a Dependent Variable: Soxtherm 8082

Table 11 Regression-Soxhlet vs. PCB-XL Daily Calibration, Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.913(a)	.834	.831	2.06007

a Predictors: (Constant), XL Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1175.126	1	1175.126	276.900	.000(a)
	Residual	233.413	55	4.244		
	Total	1408.539	56			

a Predictors: (Constant), XL Daily Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.565	.321		1.763	.083
	XL Daily Cal	.927	.056	.913	16.640	.000

a Dependent Variable: Soxhlet 8082

Table 12 Regression-Soxtherm vs. PCB-XL Daily Calibration, Complete Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.912(a)	.832	.829	2.74672

a Predictors: (Constant), XL Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	2058.764	1	2058.764	272.884	.000(a)
	Residual	414.945	55	7.544		
	Total	2473.710	56			

a Predictors: (Constant), XL Daily Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.515	.427		1.205	.234
	XL Daily Cal	1.227	.074	.912	16.519	.000

a Dependent Variable: Soxtherm 8082

Table 13 Regression-Soxtherm vs. PCB-XL Daily Calibration, Complete Data Set

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	Soxhlet 8082	.8972	25	.90377	.18075
	Soxtherm 8082	.8743	25	.87391	.17478
Pair 2	Soxhlet 8082	.8972	25	.90377	.18075
	PCB Ave Cal	.755	25	.3855	.0771
Pair 3	Soxhlet 8082	.8972	25	.90377	.18075
	PCB Daily Cal	.8076	25	.45048	.09010
Pair 4	Soxhlet 8082	.8972	25	.90377	.18075
	XL Ave Cal	.9948	25	.84275	.16855
Pair 5	Soxhlet 8082	.8972	25	.90377	.18075
	XL Daily Cal	.9584	25	.80247	.16049
Pair 6	Soxtherm 8082	.8743	25	.87391	.17478
	PCB Ave Cal	.755	25	.3855	.0771
Pair 7	Soxtherm 8082	.8743	25	.87391	.17478
	PCB Daily Cal	.8076	25	.45048	.09010
Pair 8	Soxtherm 8082	.8743	25	.87391	.17478
	XL Ave Cal	.9948	25	.84275	.16855
Pair 9	Soxtherm 8082	.8743	25	.87391	.17478
	XL Daily Cal	.9584	25	.80247	.16049

Table 13 Regression-Soxtherm vs. PCB-XL Daily Calibration, Complete Data Set (Continued)

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	Soxhlet 8082 & Soxtherm 8082	25	.981	.000
Pair 2	Soxhlet 8082 & PCB Ave Cal	25	.775	.000
Pair 3	Soxhlet 8082 & PCB Daily Cal	25	.844	.000
Pair 4	Soxhlet 8082 & XL Ave Cal	25	.471	.017
Pair 5	Soxhlet 8082 & XL Daily Cal	25	.397	.050
Pair 6	Soxtherm 8082 & PCB Ave Cal	25	.771	.000
Pair 7	Soxtherm 8082 & PCB Daily Cal	25	.839	.000
Pair 8	Soxtherm 8082 & XL Ave Cal	25	.454	.023
Pair 9	Soxtherm 8082 & XL Daily Cal	25	.380	.061

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	Soxhlet 8082 - Soxtherm 8082	.0228	.17420	.03484	-.0491	.0947	.656	24	.518
Pair 2	Soxhlet 8082 - PCB Ave Cal	.1420	.65239	.13048	-.1273	.4113	1.088	24	.287
Pair 3	Soxhlet 8082 - PCB Daily Cal	.0896	.57666	.11533	-.1485	.3276	.777	24	.445
Pair 4	Soxhlet 8082 - XL Ave Cal	-.0976	.89945	.17989	-.4689	.2736	-.543	24	.592
Pair 5	Soxhlet 8082 - XL Daily Cal	-.0612	.94087	.18817	-.4496	.3271	-.325	24	.748
Pair 6	Soxtherm 8082 - PCB Ave Cal	.1191	.62702	.12540	-.1397	.3779	.950	24	.352
Pair 7	Soxtherm 8082 - PCB Daily Cal	.0667	.55326	.11065	-.1617	.2951	.603	24	.552
Pair 8	Soxtherm 8082 - XL Ave Cal	-.1205	.89767	.17953	-.4910	.2501	-.671	24	.509
Pair 9	Soxtherm 8082 - XL Daily Cal	-.0841	.93502	.18700	-.4700	.3019	-.450	24	.657

Table 14 Regression-Soxhlet vs. Soxtherm on >0.011 and <3.0mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	Soxtherm 8082(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.981(a)	.963	.961	.17745

a Predictors: (Constant), Soxtherm 8082

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	18.879	1	18.879	599.568	.000(a)
	Residual	.724	23	.031		
	Total	19.603	24			

a Predictors: (Constant), Soxtherm 8082
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.010	.051		.194	.848
	Soxtherm 8082	1.015	.041	.981	24.486	.000

a Dependent Variable: Soxhlet 8082

Table 15 Regression-Soxhlet vs. PCB Average Calibration on >0.011 and <3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.775(a)	.600	.583	.58380

a Predictors: (Constant), PCB Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	11.764	1	11.764	34.518	.000(a)
	Residual	7.839	23	.341		
	Total	19.603	24			

a Predictors: (Constant), PCB Ave Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	-.474	.261		-1.818	.082
	PCB Ave Cal	1.816	.309	.775	5.875	.000

a Dependent Variable: Soxhlet 8082

Table 16 Regression-Soxtherm vs. PCB Average Calibration, >0.011 and < 3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.771(a)	.594	.576	.56900

a Predictors: (Constant), PCB Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	10.883	1	10.883	33.613	.000(a)
	Residual	7.447	23	.324		
	Total	18.329	24			

a Predictors: (Constant), PCB Ave Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	-.445	.254		-1.749	.094
	PCB Ave Cal	1.747	.301	.771	5.798	.000

a Dependent Variable: Soxtherm 8082

Table 17 Regression-Soxhlet vs. PCB Daily Calibration on >0.011 and <3 ppm Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.844(a)	.712	.700	.49522

a Predictors: (Constant), PCB Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	13.963	1	13.963	56.934	.000(a)
	Residual	5.641	23	.245		
	Total	19.603	24			

a Predictors: (Constant), PCB Daily Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	-.470	.207		-2.277	.032
	PCB Daily Cal	1.693	.224	.844	7.545	.000

a Dependent Variable: Soxhlet 8082

Table 18 Regression-Soxtherm vs. PCB Daily Calibration on >0.011 and <3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	PCB Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.839(a)	.704	.691	.48582

a Predictors: (Constant), PCB Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	12.901	1	12.901	54.660	.000(a)
	Residual	5.429	23	.236		
	Total	18.329	24			

a Predictors: (Constant), PCB Daily Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	-.440	.203		-2.172	.040
	PCB Daily Cal	1.628	.220	.839	7.393	.000

a Dependent Variable: Soxtherm 8082

Table 19 Regression-Soxhlet vs. PCB-XL Average Calibration, >0.011 and < 3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.471(a)	.222	.188	.81422

a Predictors: (Constant), XL Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	4.355	1	4.355	6.570	.017(a)
	Residual	15.248	23	.663		
	Total	19.603	24			

a Predictors: (Constant), XL Ave Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.394	.255		1.546	.136
	XL Ave Cal	.505	.197	.471	2.563	.017

a Dependent Variable: Soxhlet 8082

Table 20 Regression-Soxtherm vs. PCB-XL Average Calibration, >0.011 and < 3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.454(a)	.206	.171	.79559

a Predictors: (Constant), XL Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	3.771	1	3.771	5.958	.023(a)
	Residual	14.558	23	.633		
	Total	18.329	24			

a Predictors: (Constant), XL Ave Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.406	.249		1.631	.116
	XL Ave Cal	.470	.193	.454	2.441	.023

a Dependent Variable: Soxtherm 8082

Table 21 Regression-Soxhlet vs. PCB-XL Daily Cal, >0.011 and < 3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.397(a)	.157	.121	.84743

a Predictors: (Constant), XL Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	3.086	1	3.086	4.297	.050(a)
	Residual	16.517	23	.718		
	Total	19.603	24			

a Predictors: (Constant), XL Daily Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.469	.267		1.755	.093
	XL Daily Cal	.447	.216	.397	2.073	.050

a Dependent Variable: Soxhlet 8082

Table 22 Regression-Soxtherm vs. PCB-XL Daily Calibration, >0.011 and < 3.0 mg/kg Data Set

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	XL Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxtherm 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.380(a)	.145	.107	.82563

a Predictors: (Constant), XL Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	2.651	1	2.651	3.889	.061(a)
	Residual	15.678	23	.682		
	Total	18.329	24			

a Predictors: (Constant), XL Daily Cal
b Dependent Variable: Soxtherm 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.477	.260		1.834	.080
	XL Daily Cal	.414	.210	.380	1.972	.061

a Dependent Variable: Soxtherm 8082

Table 23
T-Test-Mean of Triplicates selected >0.011 and < 3.0 mg/kg

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	Soxhlet 8082	.8333	9	.92372	.30791
	Soxtherm 8082	.8111	9	.89942	.29981
Pair 2	Soxhlet 8082	.8333	9	.92372	.30791
	Hyb Ave Cal	.7267	9	.35146	.11715
Pair 3	Soxhlet 8082	.8333	9	.92372	.30791
	Hyb Daily Cal	.7756	9	.42907	.14302
Pair 4	Soxhlet 8082	.8333	9	.92372	.30791
	XL Ave Cal	.9411	9	.60654	.20218
Pair 5	Soxhlet 8082	.8333	9	.92372	.30791
	XL Daily Cal	.8922	9	.57072	.19024
Pair 6	Soxtherm 8082	.8111	9	.89942	.29981
	Hyb Ave Cal	.7267	9	.35146	.11715
Pair 7	Soxtherm 8082	.8111	9	.89942	.29981
	Hyb Daily Cal	.7756	9	.42907	.14302
Pair 8	Soxtherm 8082	.8111	9	.89942	.29981
	XL Ave Cal	.9411	9	.60654	.20218
Pair 9	Soxtherm 8082	.8111	9	.89942	.29981
	XL Daily Cal	.8922	9	.57072	.19024

Table 23
T-Test-Mean of Triplicates selected >0.011 and < 3.0 mg/kg (Continued)

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	Soxhlet 8082 & Soxtherm 8082	9	.999	.000
Pair 2	Soxhlet 8082 & Hyb Ave Cal	9	.894	.001
Pair 3	Soxhlet 8082 & Hyb Daily Cal	9	.937	.000
Pair 4	Soxhlet 8082 & XL Ave Cal	9	.678	.045
Pair 5	Soxhlet 8082 & XL Daily Cal	9	.611	.081
Pair 6	Soxtherm 8082 & Hyb Ave Cal	9	.897	.001
Pair 7	Soxtherm 8082 & Hyb Daily Cal	9	.937	.000
Pair 8	Soxtherm 8082 & XL Ave Cal	9	.690	.040
Pair 9	Soxtherm 8082 & XL Daily Cal	9	.623	.073

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	Soxhlet 8082 - Soxtherm 8082	.0222	.04353	.01451	-.0112	.0557	1.532	8	.164
Pair 2	Soxhlet 8082 - Hyb Ave Cal	.1067	.62954	.20985	-.3772	.5906	.508	8	.625
Pair 3	Soxhlet 8082 - Hyb Daily Cal	.0578	.54272	.18091	-.3594	.4749	.319	8	.758
Pair 4	Soxhlet 8082 - XL Ave Cal	-.1078	.67965	.22655	-.6302	.4146	-.476	8	.647
Pair 5	Soxhlet 8082 - XL Daily Cal	-.0589	.73131	.24377	-.6210	.5032	-.242	8	.815
Pair 6	Soxtherm 8082 - Hyb Ave Cal	.0844	.60459	.20153	-.3803	.5492	.419	8	.686
Pair 7	Soxtherm 8082 - Hyb Daily Cal	.0356	.51950	.17317	-.3638	.4349	.205	8	.842
Pair 8	Soxtherm 8082 - XL Ave Cal	-.1300	.65123	.21708	-.6306	.3706	-.599	8	.566
Pair 9	Soxtherm 8082 - XL Daily Cal	-.0811	.70346	.23449	-.6218	.4596	-.346	8	.738

Table 24 Regression-Soxhlet vs. Soxtherm, Mean Data Set with Data >0.011 and < 3.0 mg/kg

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	Soxtherm 8082(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.999(a)	.998	.998	.03912

a Predictors: (Constant), Soxtherm 8082

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	6.815	1	6.815	4453.561	.000(a)
	Residual	.011	7	.002		
	Total	6.826	8			

a Predictors: (Constant), Soxtherm 8082
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	.001	.018		.054	.959
	Soxtherm 8082	1.026	.015	.999	66.735	.000

a Dependent Variable: Soxhlet 8082

Table 25 Regression-Soxhlet vs. PCB Daily Calibration, Mean Data, >0.011 and < 3.0 mg/kg

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	Hyb Daily Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.937(a)	.878	.861	.34474

a Predictors: (Constant), Hyb Daily Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	5.994	1	5.994	50.436	.000(a)
	Residual	.832	7	.119		
	Total	6.826	8			

a Predictors: (Constant), Hyb Daily Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	-.731	.248		-2.943	.022
	Hyb Daily Cal	2.017	.284	.937	7.102	.000

a Dependent Variable: Soxhlet 8082

Table 26 Regression-Soxhlet vs. PCB Average Calibration, Mean Data Set with Data >0.011 and < 3.0 mg/kg

Variables Entered/Removed(b)

Model	Variables Entered	Variables Removed	Method
1	Hyb Ave Cal(a)	.	Enter

a All requested variables entered.
b Dependent Variable: Soxhlet 8082

Model Summary

Model	R	R Square	Adjusted R Square	Std. Error of the Estimate
1	.894(a)	.799	.770	.44254

a Predictors: (Constant), Hyb Ave Cal

ANOVA(b)

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	5.455	1	5.455	27.855	.001(a)
	Residual	1.371	7	.196		
	Total	6.826	8			

a Predictors: (Constant), Hyb Ave Cal
b Dependent Variable: Soxhlet 8082

Coefficients(a)

Model		Unstandardized Coefficients		Standardized Coefficients	t	Sig.
		B	Std. Error	Beta		
1	(Constant)	-.874	.356		-2.458	.044
	Hyb Ave Cal	2.350	.445	.894	5.278	.001

a Dependent Variable: Soxhlet 8082

Table 27 T-Test-Wet vs Dry with All Data <3.0 mg/kg

Paired Samples Statistics

		Mean	N	Std. Deviation	Std. Error Mean
Pair 1	Soxhlet	1.7083	12	.62734	.18110
	Soxtherm	1.6492	12	.62980	.18181
Pair 2	Soxhlet	1.7083	12	.62734	.18110
	PCB Ave Cal	1.1075	12	.20222	.05838
Pair 3	Soxhlet	1.7083	12	.62734	.18110
	PCB Daily Cal	1.1608	12	.21669	.06255
Pair 4	Soxhlet	1.7083	12	.62734	.18110
	PCB 1 ppm	1.5833	12	.51493	.14865
Pair 5	Soxhlet	1.7083	12	.62734	.18110
	PCB Ave Wet	.8592	12	.21948	.06336
Pair 6	Soxhlet	1.7083	12	.62734	.18110
	PCB Daily Wet	.8192	12	.20268	.05851
Pair 7	Soxhlet	1.7083	12	.62734	.18110
	PCB 1 ppm Wet	1.3333	12	.49237	.14213
Pair 8	Soxhlet	1.7083	12	.62734	.18110
	XL Ave Cal	1.3042	12	.42217	.12187
Pair 9	Soxhlet	1.7083	12	.62734	.18110
	XL Daily Cal	1.1883	12	.35383	.10214
Pair 10	Soxhlet	1.7083	12	.62734	.18110
	XL 1 ppm	1.9167	12	.28868	.08333
Pair 11	Soxhlet	1.7083	12	.62734	.18110
	XL Ave Wet	.8033	12	.30729	.08871
Pair 12	Soxhlet	1.7083	12	.62734	.18110
	XL Daily Wet	.8483	12	.35424	.10226
Pair 13	Soxhlet	1.7083	12	.62734	.18110
	XL 1 ppm Wet	1.2500	12	.45227	.13056

Table 27 T-Test-Wet vs Dry with All Data <3.0 mg/kg (Continued)

Paired Samples Correlations

		N	Correlation	Sig.
Pair 1	Soxhlet & Soxtherm	12	.925	.000
Pair 2	Soxhlet & PCB Ave Cal	12	.919	.000
Pair 3	Soxhlet & PCB Daily Cal	12	.921	.000
Pair 4	Soxhlet & PCB 1 ppm	12	.715	.009
Pair 5	Soxhlet & PCB Ave Wet	12	.891	.000
Pair 6	Soxhlet & PCB Daily Wet	12	.889	.000
Pair 7	Soxhlet & PCB 1 ppm Wet	12	.814	.001
Pair 8	Soxhlet & XL Ave Cal	12	.824	.001
Pair 9	Soxhlet & XL Daily Cal	12	.825	.001
Pair 10	Soxhlet & XL 1 ppm	12	.305	.334
Pair 11	Soxhlet & XL Ave Wet	12	.846	.001
Pair 12	Soxhlet & XL Daily Wet	12	.844	.001
Pair 13	Soxhlet & XL 1 ppm Wet	12	.761	.004

Table 27 T-Test-Wet vs Dry with All Data <3.0 mg/kg (Continued)

Paired Samples Test

		Paired Differences					t	df	Sig. (2-tailed)
		Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
					Lower	Upper			
Pair 1	Soxhlet - Soxtherm	.0592	.24310	.07018	-.0953	.2136	.843	11	.417
Pair 2	Soxhlet - PCB Ave Cal	.6008	.44868	.12952	.3158	.8859	4.639	11	.001
Pair 3	Soxhlet - PCB Daily Cal	.5475	.43587	.12583	.2706	.8244	4.351	11	.001
Pair 4	Soxhlet - PCB 1 ppm	.1250	.44339	.12799	-.1567	.4067	.977	11	.350
Pair 5	Soxhlet - PCB Ave Wet	.8492	.44322	.12795	.5676	1.1308	6.637	11	.000
Pair 6	Soxhlet - PCB Daily Wet	.8892	.45676	.13185	.5990	1.1794	6.744	11	.000
Pair 7	Soxhlet - PCB 1 ppm Wet	.3750	.36463	.10526	.1433	.6067	3.563	11	.004
Pair 8	Soxhlet - XL Ave Cal	.4042	.36761	.10612	.1706	.6377	3.809	11	.003
Pair 9	Soxhlet - XL Daily Cal	.5200	.39057	.11275	.2718	.7682	4.612	11	.001
Pair 10	Soxhlet - XL 1 ppm	-.2083	.60522	.17471	-.5929	.1762	-1.192	11	.258
Pair 11	Soxhlet - XL Ave Wet	.9050	.40232	.11614	.6494	1.1606	7.792	11	.000
Pair 12	Soxhlet - XL Daily Wet	.8600	.37938	.10952	.6190	1.1010	7.853	11	.000
Pair 13	Soxhlet - XL 1 ppm Wet	.4583	.40778	.11772	.1992	.7174	3.894	11	.003

Table 28
PCB Data by Aroclor and Total PCB for Methods Showing Best Correlation (Continued)

Sample No.	Replicate No.	Soxhlet 8082 Total PCB (mg/kg)	Soxtherm 8082 Total PCB (mg/kg)	PCB Daily Cal Total PCB (mg/kg)	Soxhlet 8082 PCB 1242 (mg/kg)	Soxhlet 8082 PCB 1254 (mg/kg)
1	1	2.1	2.3	1.27	2.1	0.011
1	10	1.8	1.8	1.29	1.8	0.011
1	15	2	1.7	1.24	2	0.011
2	2	12	16	6.69	12	0.011
2	8	12	14	7.09	12	0.011
2	21	12	16	6.78	12	0.011
3	3	12	16	6.78	12	0.011
3	13	12	16	7.76	12	0.011
3	17	12	16	7.98	12	0.011
4	4	3.3	3.2	1.4	3.3	0.011
4	11	3.2	3.3	1.35	3.2	0.011
4	20	3.4	2.6	1.27	3.4	0.011
5	5	1.5	1.2	0.98	1.5	0.011
5	12	1.1	1.2	1.08	1.1	0.011
5	16	1.2	1.1	0.96	1.2	0.011
6	6	1.1	0.99	1.01	1.1	0.011
6	14	1.1	1.1	0.92	1.1	0.011
6	19	1.1	1	0.87	1.1	0.011
7	7	2.6	2.3	1.5	2.6	0.011
7	9	2	2.5	1.37	2	0.011
7	18	2.9	2.6	1.44	2.9	0.011
8	22	0.011	0.01	0.19	0.011	0.011
8	39	0.011	0.011	0.18	0.011	0.011
8	54	0.011	0.011	0.49	0.011	0.011
9	23	18	22.8	20	16	2
9	45	17.9	21.7	17	16	1.9
9	49	15.7	23.7	17	14	1.7
10	24	0.011	0.011	0.385	0.011	0.011
10	38	0.011	0.048	0.33	0.011	0.011
10	51	0.025	0.011	0.2	0.025	0.011
11	25	0.039	0.066	0.19	0.039	0.011
11	44	0.075	0.11	0.18	0.075	0.011
11	52	0.054	0.068	0.2	0.054	0.011
12	26	0.16	0.16	0.42	0.029	0.011
12	37	0.095	0.11	0.53	0.095	0.011
12	50	0.134	0.09	0.54	0.1	0.034
13	27	8	10.1	4.9	7	1
13	43	7.4	9.3	5.4	6	1.4
13	46	6.29	9.8	3.7	5.4	0.89
14	28	0.011	0.011	0.19	0.011	0.011
14	36	0.011	0.011	0.48	0.011	0.011
14	47	0.011	0.011	0.18	0.011	0.011
15	29	5.75	7.1	2.7	4.8	0.95
15	42	4.83	5.02	3.7	3.9	0.93
15	48	3.51	5.99	3.5	2.8	0.71
16	30	0.224	0.28	0.48	0.14	0.084
16	35	0.16	0.24	0.45	0.099	0.065
16	57	0.28	0.26	1.53	0.17	0.11
17	31	0.011	0.011	0.19	0.011	0.011
17	41	0.011	0.011	0.18	0.011	0.011
17	56	0.011	0.011	0.59	0.011	0.011
18	32	0.27	0.13	0.49	0.23	0.04
18	34	0.2	0.323	0.41	0.17	0.031

Table 28
PCB Data by Aroclor and Total PCB for Methods Showing Best Correlation (Continued)

Sample No.	Replicate No.	Soxhlet 8082 Total PCB (mg/kg)	Soxtherm 8082 Total PCB (mg/kg)	PCB Daily Cal Total PCB (mg/kg)	Soxhlet 8082 PCB 1242 (mg/kg)	Soxhlet 8082 PCB 1254 (mg/kg)
18	53	0.213	0.22	0.64	0.18	0.033
19	33	0.011	0.011	0.4	0.011	0.011
19	40	0.011	0.011	0.18	0.011	0.011
19	55	0.011	0.011	0.21	0.011	0.011

Method Detection Limit (MDL) for 8082 is 0.022 mg/kg, where data was a nondetect half of the MDL, 0.011 mg/kg was used for statistical assessment of the data point.
Reporting Limit (RL) for Hybrizyme ranged from 0.38 to 0.50 mg/kg, where data was nondetect half of the RL was used for the statistical assessment of the data point.

Table 29
Fox River Sediment Ultrasonic Bath Study

		Hybrizyme	PCB Daily Calibration Curve			Hybrizyme	PCB Average Calibration Curve		
		(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
Sample No.	Replicate No.	0 min.	15 min.	30 min.	45 min.	0 min.	15 min.	30 min.	45 min.
2	2	4.1	3.9	3.7	4.0	3.6	3.5	3.3	3.5
2	8	4.1	4.0	4.1	4.1	3.7	3.6	3.7	3.6
3	3	4.6	4.5	4.5	5.1	4.2	4.1	4.0	4.6
4	4	1.4	1.4	1.5	1.3	1.2	1.2	1.3	1.1
4	11	1.4	1.4	1.5	1.6	1.3	1.3	1.3	1.4
4	20	1.4	1.3	1.3	1.5	1.3	1.1	1.2	1.3
7	9	1.5	1.6	1.7	1.6	1.3	1.4	1.5	1.4
7	18	1.5	1.4	1.6	1.7	1.3	1.3	1.4	1.5
7	18 duplicate	1.4	1.5	1.5	1.5	1.3	1.4	1.3	1.3
	MB	<0.48	<0.48	<0.48	<0.48	<0.41	<0.41	<0.41	<0.41
	LCS	1.1	1.1	1	0.92	1	0.91	0.89	0.8

Table 30
Mean of Total PCB Data of Triplicates for 8082 and Hybrizyme Protocols by Calibration Routine

Sample No.	Soxhlet 8082 Total PCB (mg/kg)	Soxtherm 8082 Total PCB (mg/kg)	PCB Ave Cal Total PCB (mg/kg)	PCB Daily Cal Total PCB (mg/kg)	PCB-XL Ave Cal Total PCB (mg/kg)	PCB-XL Daily Cal Total PCB (mg/kg)
1	1.97	1.93	1.11	1.27	1.23	1.16
2	12	15.33	6.23	6.85	7.82	6.57
3	12	16	6.8	7.51	8.81	7.34
4	3.3	3.03	1.18	1.34	1.27	1.19
5	1.27	1.17	0.88	1.01	1.15	1.09
6	1.1	1.03	0.81	0.93	0.9	0.88
7	2.5	2.47	1.27	1.44	1.85	1.62
8	0.011	0.011	0.31	0.29	0.39	0.41
9	17.2	22.73	19.33	18	23	21
10	0.02	0.02	0.33	0.31	0.26	0.28
11	0.06	0.08	0.21	0.19	0.33	0.39
12	0.13	0.12	0.53	0.5	0.43	0.44
13	7.23	9.73	4.8	4.67	7.13	6.83
14	0.01	0.01	0.32	0.28	0.3	0.35
15	4.7	6.04	3.3	3.3	5.17	4.9
16	0.22	0.26	0.86	0.82	1.79	1.81
17	0.01	0.01	0.34	0.29	0.43	0.44
18	0.23	0.22	0.54	0.51	0.53	0.44
19	0.01	0.01	0.32	0.26	0.22	0.2

Table 31
Summary of Blanks, Laboratory Control Spikes (LCS) and Duplicates

	PCB Protocol						PCB-XL Protocol					
	Daily Curve			Average Curve			Daily Curve			Average Curve		
Analysis Date	Method Blk (mg/kg)	LCS (% Rec.)	Duplicate (% Agree)	Method Blk (mg/kg)	LCS (% Rec.)	Duplicate (% Agree)	Method Blk (mg/kg)	LCS (% Rec.)	Duplicate (% Agree)	Method Blk (mg/kg)	LCS (% Rec.)	Duplicate (% Agree)
5/8/2003	<0.45	90%										
5/15/2003	<0.45	96%		<0.46	101%		<0.37	94%		<0.38	89%	
5/19/2003	<0.47	95%		<0.41	85%		<0.37	93%		<0.44	98%	
6/3/2003	<0.38	86%	97%	<0.41	88%	98%	<0.42	97%	NC	<0.44	86%	NC
6/4/2003	<0.36	81%	NC	<0.41	89%	NC	<0.39	80%	NC	<0.44	90%	NC
6/5/2003	<0.40	95%	NC	<0.41	97%	NC	<0.38	98%	NC	<0.44	102%	NC
6/9/2003	<0.48*	102%*	97%*	<0.41*	89%*	97%*						
6/11/2003	<0.50	122%	95%	<0.43	104%	96%						
6/11/2003			NC			NC						

LCS are spiked Ottawa Sand at 1.0 mg/kg Aroclor 1242

NC = Not able to calculate. One or both values below detection limit.

* = Data from Ultrasonic bath study values are the average from the four extraction times (0, 15, 30 and 45 minutes).

Table 32
Fox River Sediment Matrix Spike/Matrix Spike Duplicate (MS/MSD) Study

Sample No.	Replicate No.	Sample Total PCB (mg/kg)	MS Total PCB (mg/kg)	% Recovery	MSD. Total PCB (mg/kg)	% Recovery
19	33	<0.44	1.00	100	1.03	103
14	36	<0.44	0.93	93	0.74	74
8	39	<0.44	0.96	96	0.92	92
19	40	<0.44	0.74	74	0.94	94
17	41	<0.44	0.98	98	1.03	103
14	47	<0.44	0.84	84	0.79	79
8	54	<0.44	0.90	90	0.88	88
	Method Blk	<0.44				
	LCS	1.10		110		

Table 33
Comparison of Hybrizyme Data by Protocol for Aroclors 1254 and 1260 Determined from the Aroclor 1242 Curve

PCB Protocol

Aroclor 1242		Aroclor 1254			Aroclor 1260		
Standard (ug/L)	Analyzed (ug/L)	Standard (ug/L)	Analyzed (ug/L)	Multiple of 1242	Standard (ug/L)	Analyzed (ug/L)	Multiple of 1242
100	120	25	90	3.6	30	<77	-
200	200	50	130	2.6	60	78	1.3
300	290	75	210	2.8	90	150	1.7
460	510	100	270	2.7	120	210	1.8
600	640	150	400	2.7	180	320	1.8

PCB-XL Protocol

Aroclor 1242		Aroclor 1254			Aroclor 1260		
Standard (ug/L)	Analyzed (ug/L)	Standard (ug/L)	Analyzed (ug/L)	Multiple of 1242	Standard (ug/L)	Analyzed (ug/L)	Multiple of 1242
100	110	25	120	4.8	30	150	5.0
200	200	50	250	5.0	60	330	5.5
300	310	75	450	6.0	90	770	8.6
460	500	100	710	7.1	120	>830	-
600	670	150	>830	-	180	>830	-

Table 34
Analysis of Replicates for Confirmation Rate Recommendation

Sample No.	Replicate No.	8082 Soxhlet (mg/kg)	8082 Soxtherm (mg/kg)	Hybrizyme PCB Ave (mg/kg)	Hybrizyme PCB Daily (mg/kg)	Hybrizyme 1	PCB Daily Replicate 2	(mg/kg) 3	Soxhlet 8082 Range Acceptable (mg/kg)	(mg/kg)	% Correct
1	1	2.1	2.3	1.11	1.27				0.66383	2.69639	
2	2	12	16	3.48	3.84				3.82236	8.23988	
3	3	12	16	3.74	4.16				4.21564	8.93012	
4	4	3.3	3.2	1.23	1.4				0.8236	2.9768	
5	5	1.5	1.2	0.86	0.98	1.3	0.98	1.1	0.30742	2.07086	100
6	6	1.1	0.99	0.88	1.01	1.1	1	1.1	0.34429	2.13557	100
7	7	2.6	2.3	1.33	1.5				0.9465	3.1925	
2	8	12	14	3.27	3.62				3.55198	7.76534	
7	9	2	2.5	1.21	1.37				0.78673	2.91209	
1	10	1.8	1.8	1.14	1.29	1.5	1.3		0.68841	2.73953	100
4	11	3.2	3.3	1.19	1.35				0.76215	2.86895	
5	12	1.1	1.2	0.95	1.08	1.2	1.1	0.86	0.43032	2.28656	100
3	13	12	16	3.5	3.89				3.88381	8.34773	
6	14	1.1	1.1	0.8	0.92	1.1		0.91	0.23368	1.94144	100
1	15	2	1.7	1.09	1.24				0.62696	2.63168	
5	16	1.2	1.1	0.84	0.96	1.2	0.92		0.28284	2.02772	100
3	17	12	16	3.75	4.17				4.22793	8.95169	
7	18	2.9	2.6	1.28	1.44				0.87276	3.06308	
6	19	1.1	1	0.75	0.87	1	0.87		0.17223	1.83359	100
4	20	3.4	2.6	1.11	1.27				0.66383	2.69639	
2	21	12	16	3.15	3.49				3.39221	7.48493	
8	22	0.01	0.01	0.21	0.19	0.25			-0.66349	0.36683	100
9	23	18	22.8	22	20				23.683	43.097	
10	24	0.01	0.01	0.42	0.39	0.25			-0.41769	0.79823	100
11	25	0.04	0.07	0.21	0.19	0.25			-0.66349	0.36683	100
12	26	0.16	0.16	0.45	0.42	0.25			-0.38082	0.86294	100
13	27	8	10.1	5	4.9				5.1251	10.5263	
14	28	0.01	0.01	0.21	0.19	0.25			-0.66349	0.36683	100
15	29	5.75	7.1	2.5	2.7				2.4213	5.7809	
16	30	0.22	0.28	0.51	0.48	0.54			-0.30708	0.99236	100
17	31	0.01	0.01	0.21	0.19	0.25			-0.66349	0.36683	100
18	32	0.27	0.13	0.52	0.49	0.25			-0.29479	1.01393	100
19	33	0.01	0.01	0.45	0.4				-0.4054	0.8198	
18	34	0.2	0.32	0.46	0.41	0.54	0.41		-0.39311	0.84137	100
16	35	0.16	0.24	0.5	0.45	0.25	0.45		-0.34395	0.92765	100
14	36	0.01	0.01	0.54	0.48				-0.30708	0.99236	

Table 34
Analysis of Replicates for Confirmation Rate Recommendation

Sample No.	Replicate No.	8082 Soxhlet (mg/kg)	8082 Soxtherm (mg/kg)	Hybrizyme PCB Ave (mg/kg)	Hybrizyme PCB Daily (mg/kg)	Hybrizyme 1	PCB Daily Replicate 2	(mg/kg) 3	Soxhlet 8082 Range Acceptable (mg/kg)	(mg/kg)	% Correct
12	37	0.1	0.11	0.59	0.53	0.25	0.53		-0.24563	1.10021	100
10	38	0.01	0.05	0.36	0.33				-0.49143	0.66881	
8	39	0.01	0.01	0.21	0.18				-0.67578	0.34526	
19	40	0.01	0.01	0.21	0.18				-0.67578	0.34526	
17	41	0.01	0.01	0.21	0.18				-0.67578	0.34526	
15	42	4.83	5.02	3.8	3.7				3.6503	7.9379	
13	43	7.4	9.3	5.5	5.4				5.7396	11.6048	
11	44	0.08	0.11	0.21	0.18	0.25	0.18		-0.67578	0.34526	100
9	45	17.9	21.7	19	17				19.996	36.626	
13	46	6.29	9.8	4.4	3.7				3.6503	7.9379	
14	47	0.01	0.01	0.21	0.18				-0.67578	0.34526	
15	48	3.51	5.99	3.6	3.5				3.4045	7.5065	
9	49	15.7	23.7	17	17				19.996	36.626	
12	50	0.13	0.09	0.55	0.54	0.25	0.54		-0.23334	1.12178	100
10	51	0.03	0.01	0.21	0.2	0.25	0.2		-0.6512	0.3884	100
11	52	0.05	0.07	0.21	0.2	0.25	0.2		-0.6512	0.3884	100
18	53	0.21	0.22	0.65	0.64	0.25	0.64		-0.11044	1.33748	100
8	54	0.01	0.01	0.5	0.49				-0.29479	1.01393	
19	55	0.01	0.01	0.31	0.2				-0.6512	0.3884	
17	56	0.01	0.01	0.6	0.5				-0.2825	1.0355	
16	57	0.28	0.26	1.57	1.53	0.86	1.53		0.98337	3.25721	66
Simulation of values from 0.2-2.5 mg/kg					**0.2**				**-0.6512**	**0.3884**	
					0.3				**-0.5283**	**0.6041**	
					0.4				**-0.4054**	**0.8198**	
					0.5				**-0.2825**	**1.0355**	
					1				**0.332**	**2.114**	
					1.5				**0.9465**	**3.1925**	
					1.6				**1.0694**	**3.4082**	
					1.7				**1.1923**	**3.6239**	
					1.8				**1.3152**	**3.8396**	
					1.9				**1.4381**	**4.0553**	
					2				**1.561**	**4.271**	
					2.5				**2.1755**	**5.3495**	

Recommend: Hybrizyme PCB Protocol, Daily Calibration, Air-dried sediments: Confirm results >RL and <2.0 mg/kg by 8082

Table 35
Wisconsin Tissue Mills 8082 Data Subset Compared to Hybrizyme

Subset of WTM Data >0.3 and <6.0 mg/kg

Actual 8082 PCB Concentration (mg/kg)				Composition (%)			Hybrizyme
Aroclor 1242	Aroclor 1254	Aroclor 1260	Total	1242%	1254%	1260%	Calculated* Total PCB (mg/kg)
0.23	0.1	0.02	0.35	65.71	28.57	5.71	0.48
0.16	0.17	0.02	0.35	45.71	48.57	5.71	0.49
0.18	0.13	0.05	0.36	50.00	36.11	13.89	0.49
0.29	0.07	0.02	0.39	74.36	17.95	5.13	0.51
0.33	0.05	0.02	0.4	82.50	12.50	5.00	0.52
0.32	0.11	0.07	0.5	64.00	22.00	14.00	0.59
0.61	0.04	0.04	0.7	87.14	5.71	5.71	0.73
0.62	0.22	0.12	0.96	64.58	22.92	12.50	0.92
1	0.08	0.05	1.12	89.29	7.14	4.46	1.03
0.64	0.53	0.25	1.42	45.07	37.32	17.61	1.24
1.1	0.27	0.07	1.44	76.39	18.75	4.86	1.25
0.79	0.65	0.09	1.53	51.63	42.48	5.88	1.32
1.4	0.4	0.11	1.91	73.30	20.94	5.76	1.59
1.4	0.53	0.34	2.27	61.67	23.35	14.98	1.84
1.9	0.35	0.16	2.41	78.84	14.52	6.64	1.94
2.3	0.43	0.14	2.87	80.14	14.98	4.88	2.27
1.9	0.86	0.22	2.98	63.76	28.86	7.38	2.34
2.5	0.82	0.22	3.54	70.62	23.16	6.21	2.74
3.9	0.22	0.22	4.34	89.86	5.07	5.07	3.31
3.2	1.5	0.66	5.36	59.70	27.99	12.31	4.03

*Equation used: Hybrizyme=(Total 8082+.335)/1.414

Hybrizyme correctly identified all samples for 8082 confirmatory analysis based on the study recommended range of >0.5 to <2.0 mg/kg with no false positives or negatives at the 1.0 mg/kg action level.

Figure 1
Matched Pair Results on Total Data Set, mg/kg – Soxhlet vs. Hybrizyme PCB and Soxtherm

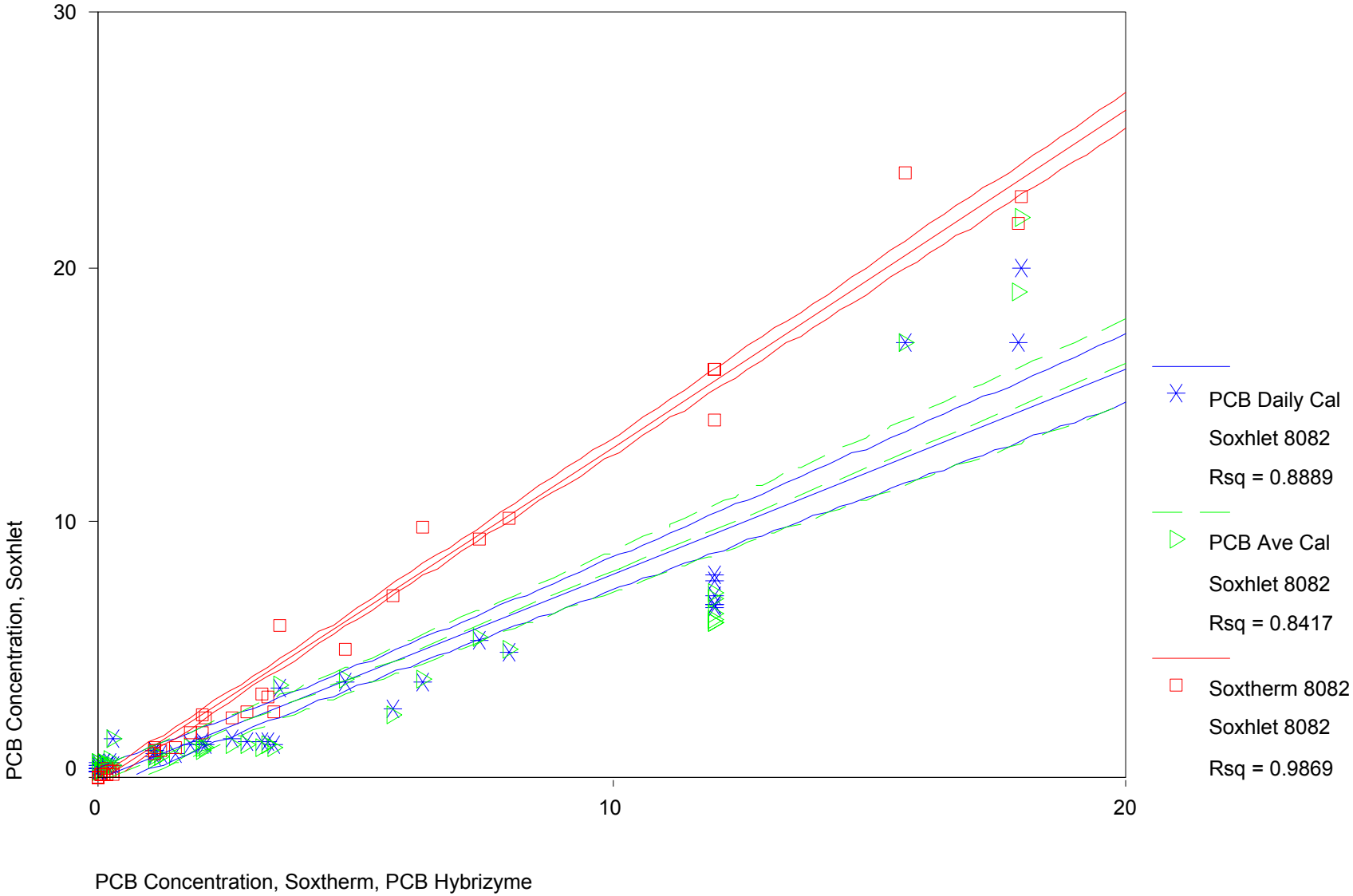

Figure 2
Matched Pair Results on Total Data Set, mg/kg – Soxhlet vs. Hybrizyme PCB-XL and Soxtherm

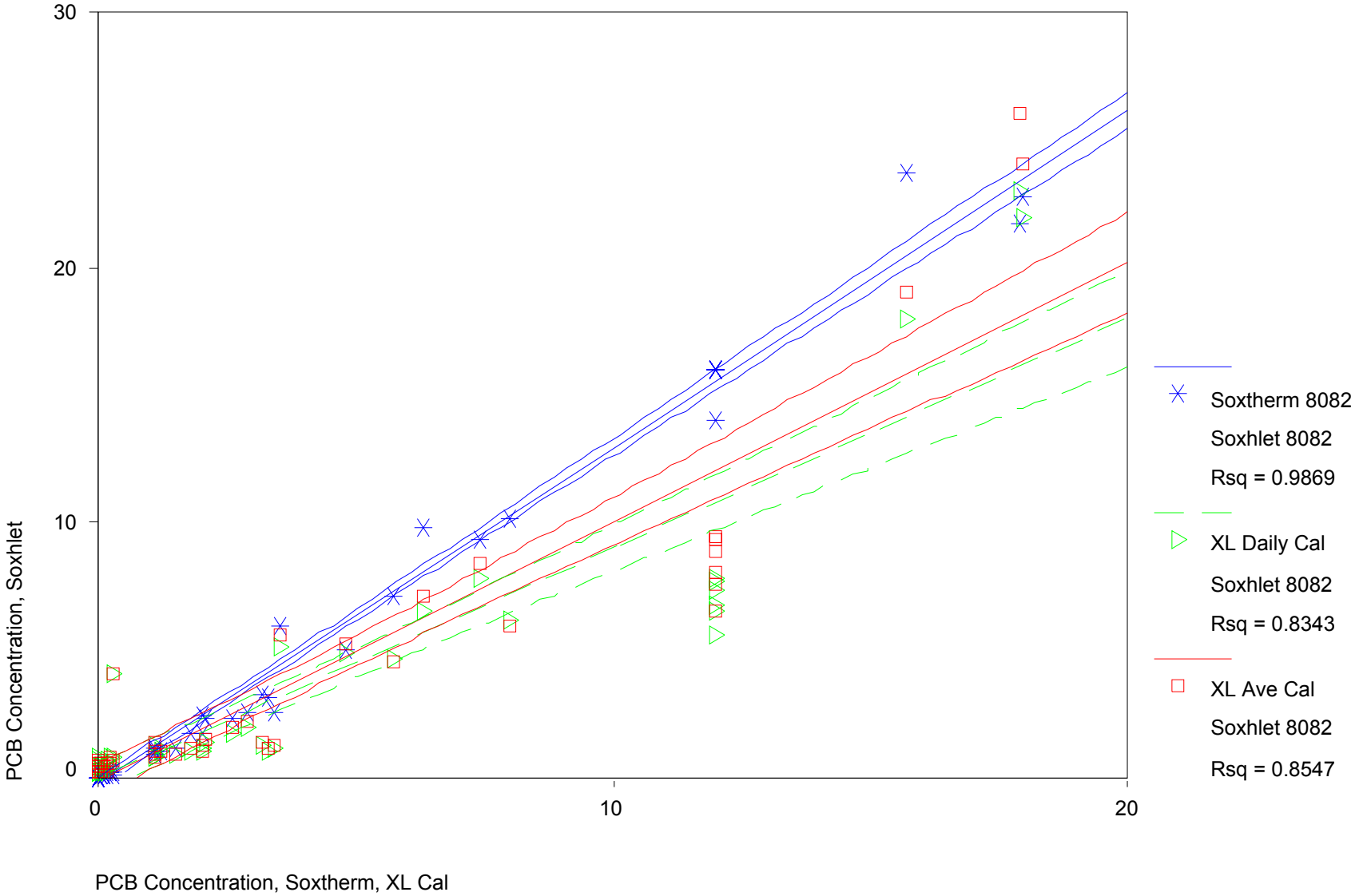

Figure 3
Matched Pair Results on Total Data Set, mg/kg – Soxtherm vs. Hybrizyme PCB and Soxhlet

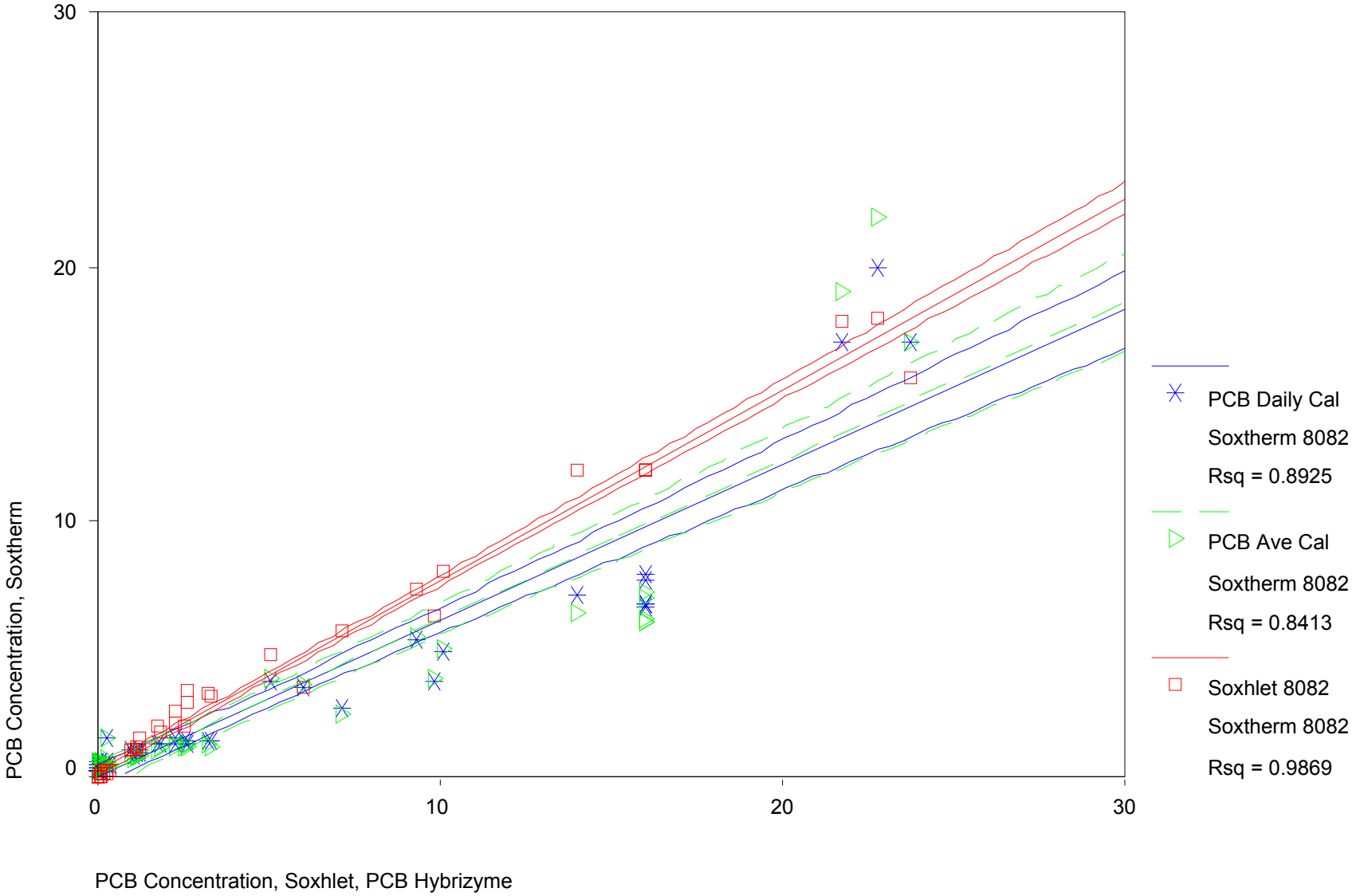

Figure 4
Matched Pair Results on Total Data Set, mg/kg – Soxtherm vs. Hybrizyme PCB-XL and Soxhlet

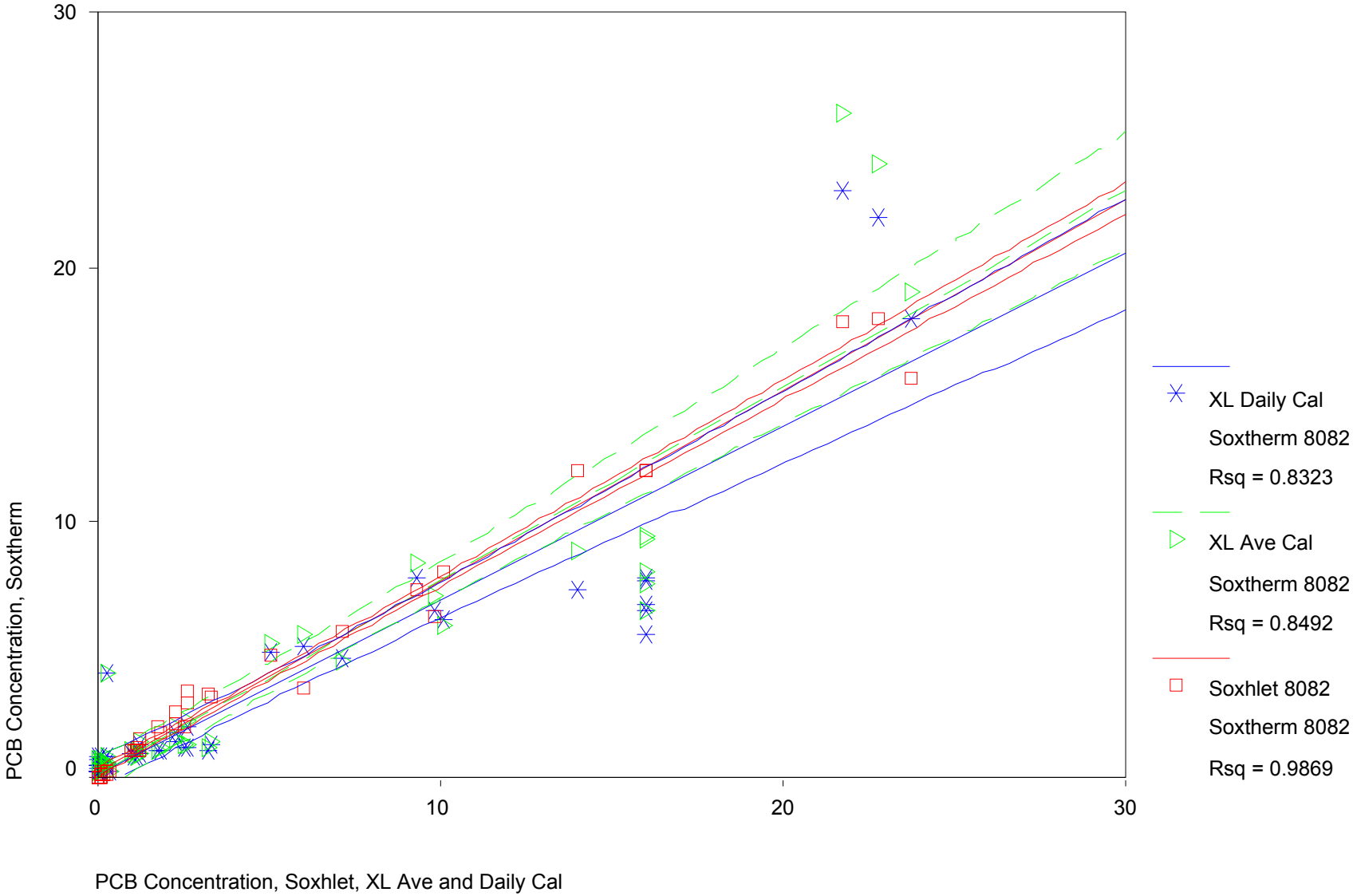

Figure 5
Matched Pair Results for >0.011 to <3.0 mg/kg Data Set – Soxhlet vs. Hybrizyme PCB and Soxtherm

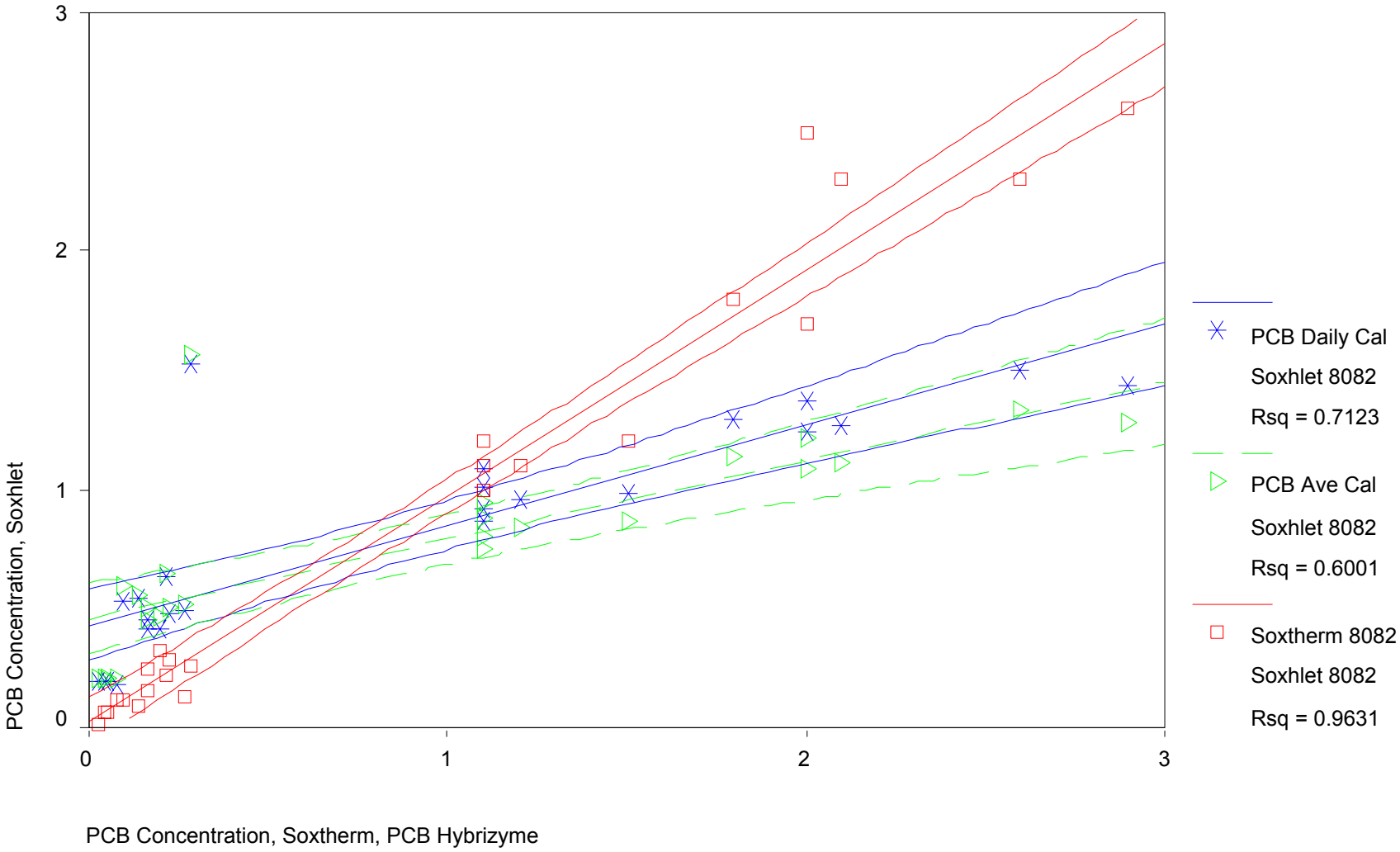

Figure 6
Matched Pair Results Results for >0.011 to <3.0 mg/kg Data Set – Soxhlet vs. Hybrizyme PCB-XL and Soxtherm

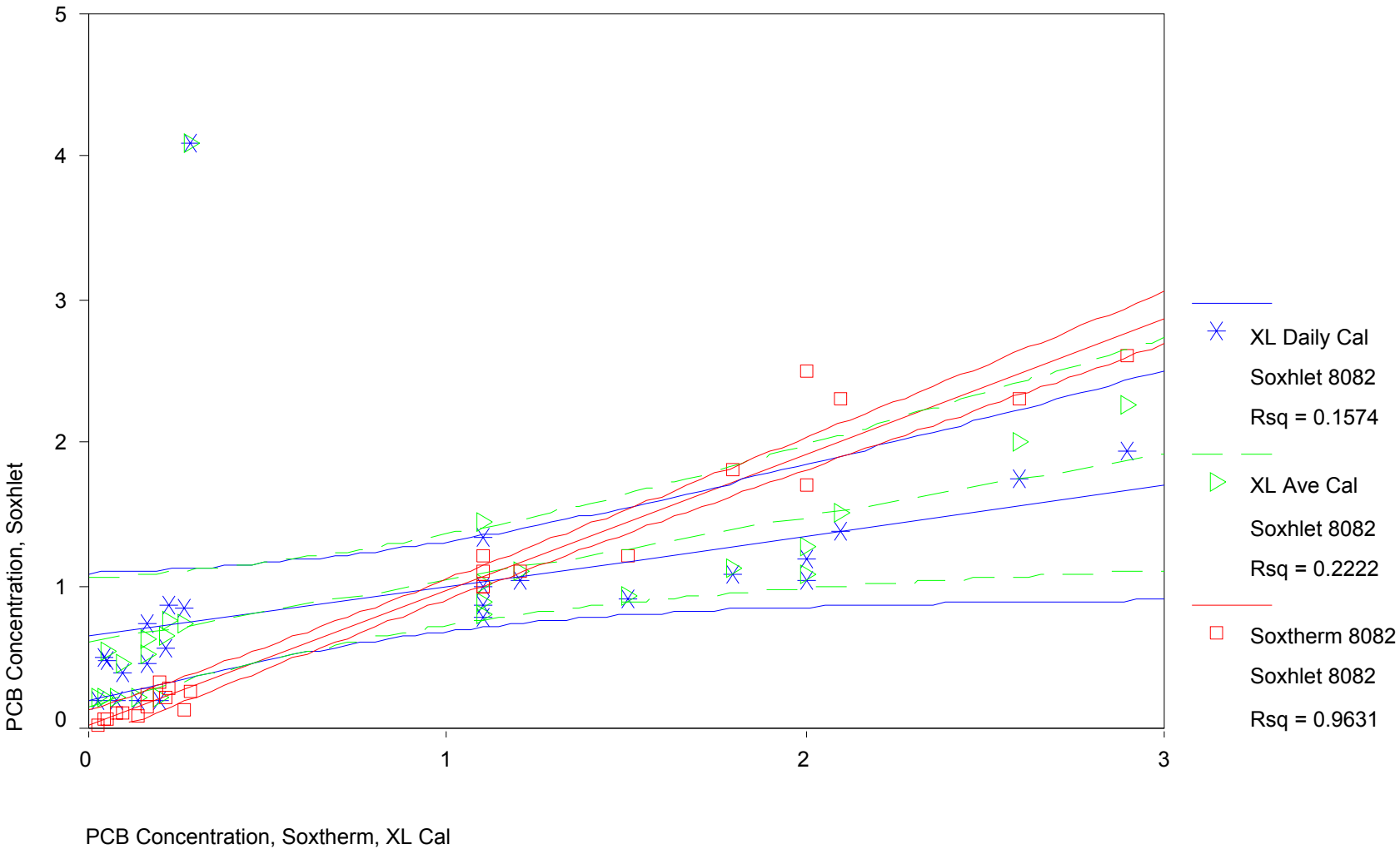

Figure 7
Matched Pair Results for >0.011 to <3.0 mg/kg Data Set – Soxtherm vs. Hybrizyme PCB and Soxhlet

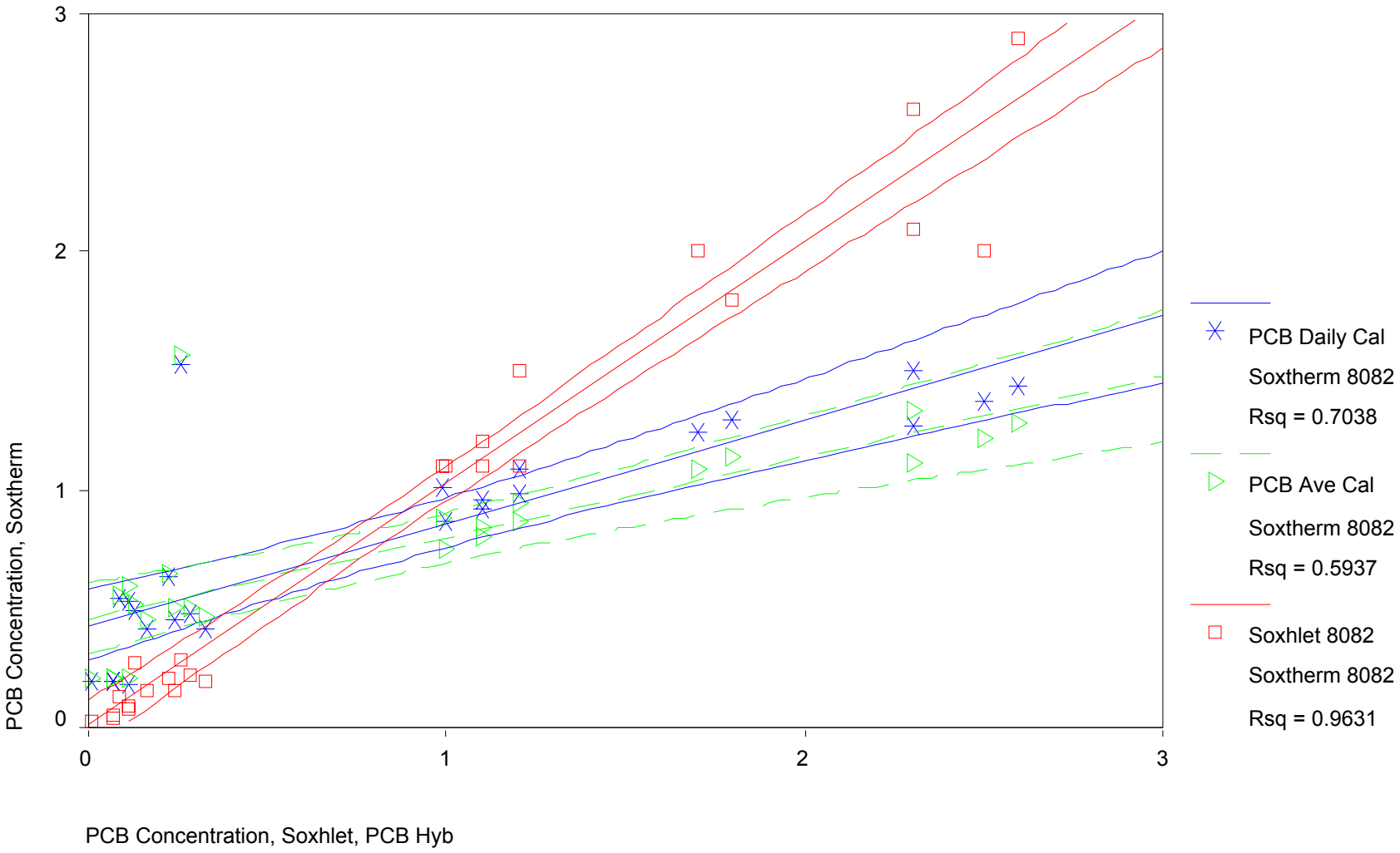

Figure 8
Matched Pair Results for >0.011 to <3.0 mg/kg Data Set – Soxtherm vs. Hybrizyme PCB-XL and Soxhlet

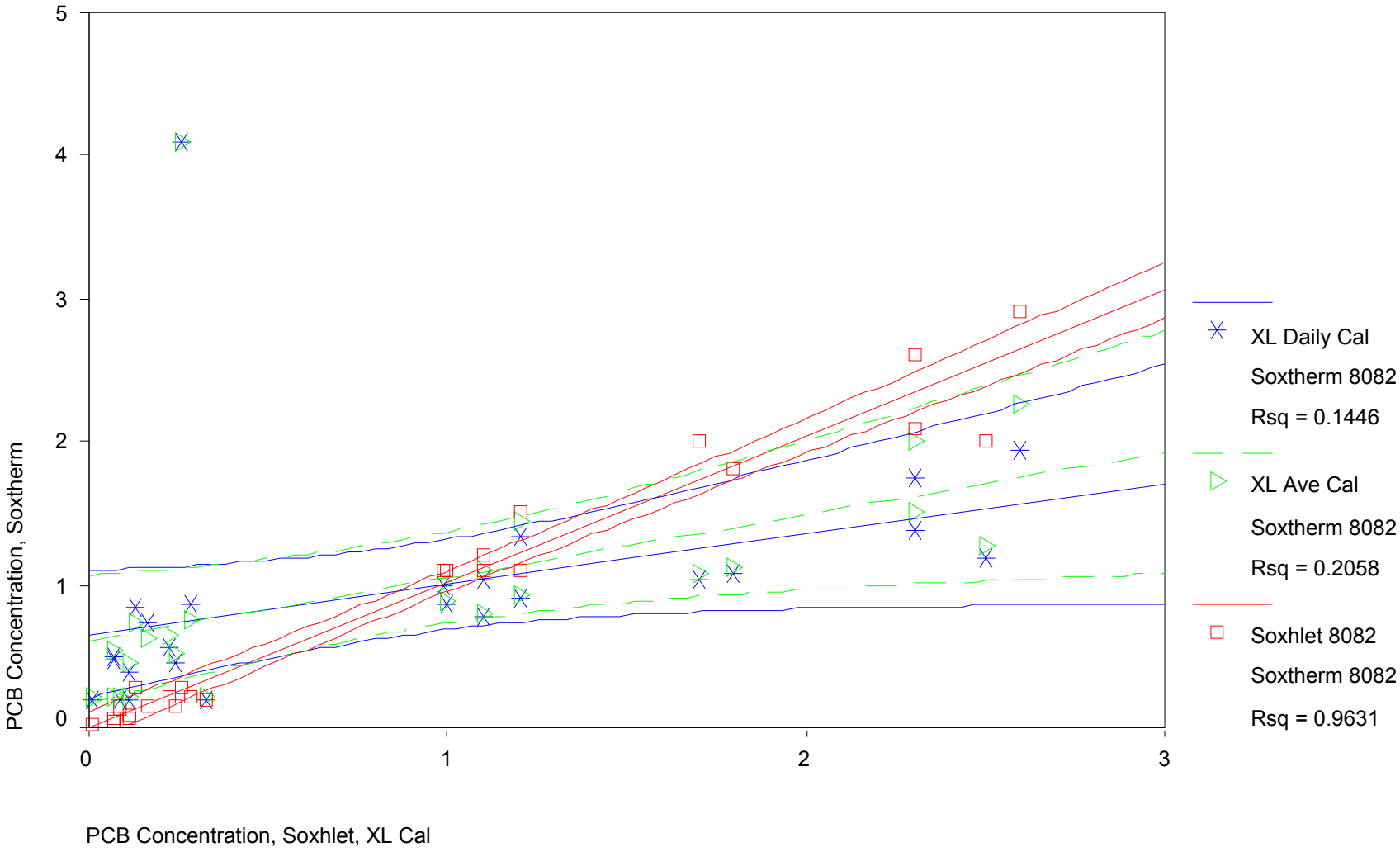

Figure 9
Mean Values of Triplicate Analyses for >0.011 to <3.0 mg/kg Data Set – Soxhlet vs. Hybrizyme PCB and Soxtherm

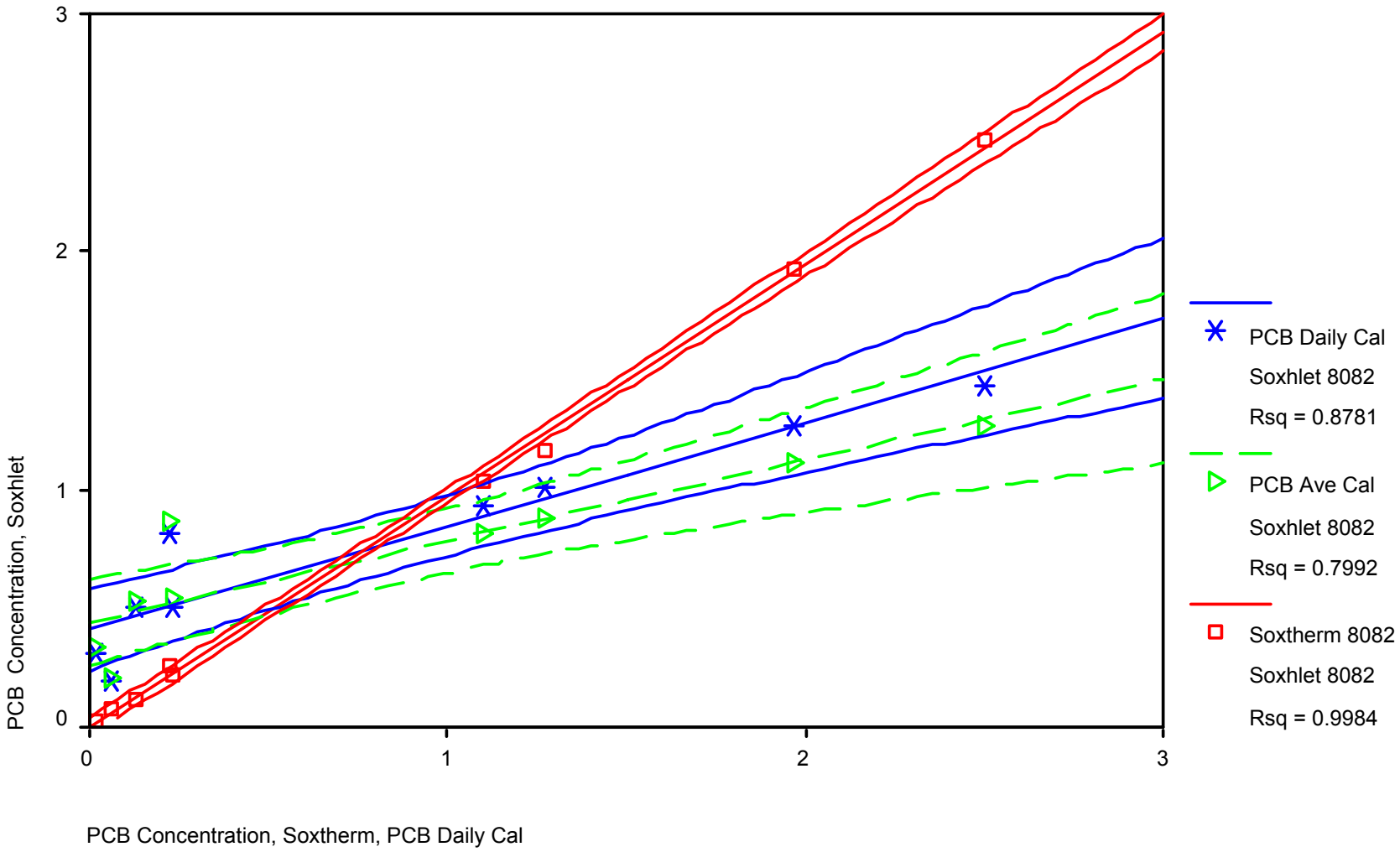

Figure 10
Mean Values of Triplicate Analyses for >0.011 to <3.0 mg/kg Data Set – Soxhlet vs. Hybrizyme PCB-XL and Soxtherm

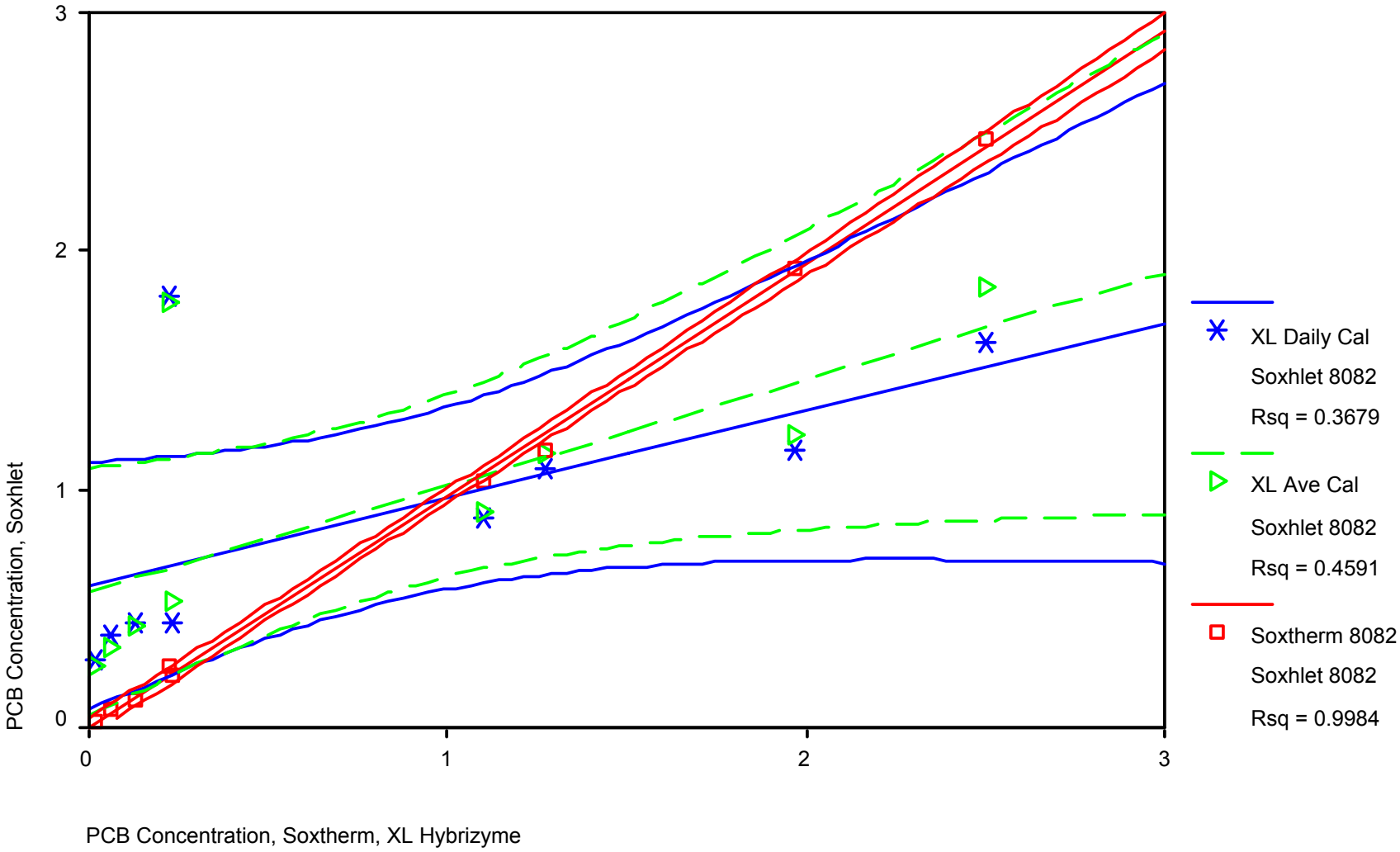

Figure 11
Mean Values of Triplicate Analyses for >0.011 to <3.0 mg/kg Data Set – Soxtherm vs. Hybrizyme PCB and Soxhlet

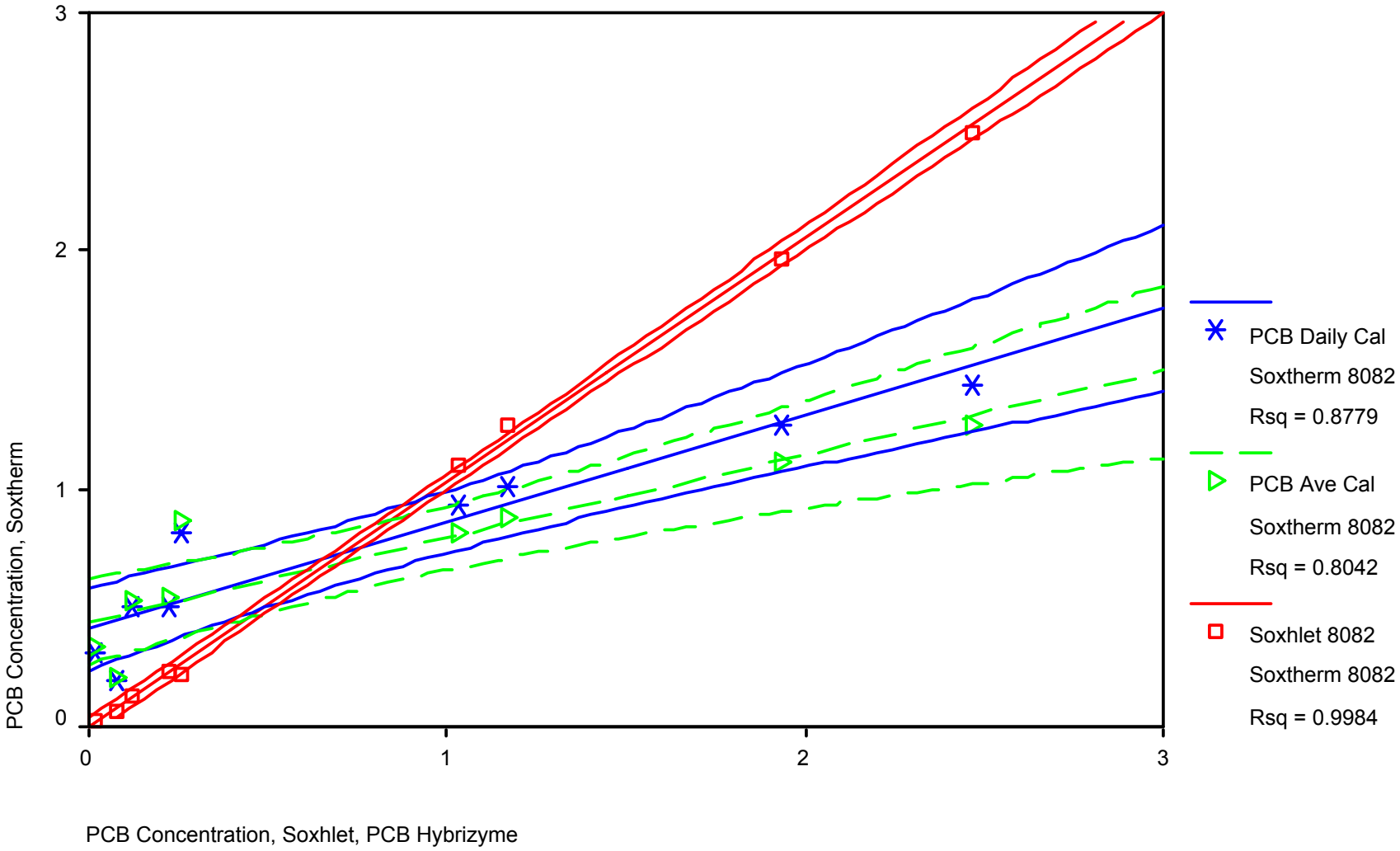

Figure 12
Mean Values of Triplicate Analyses for >0.011 to <3.0 mg/kg Data Set – Soxtherm vs. Hybrizyme PCB-XL and Soxhlet

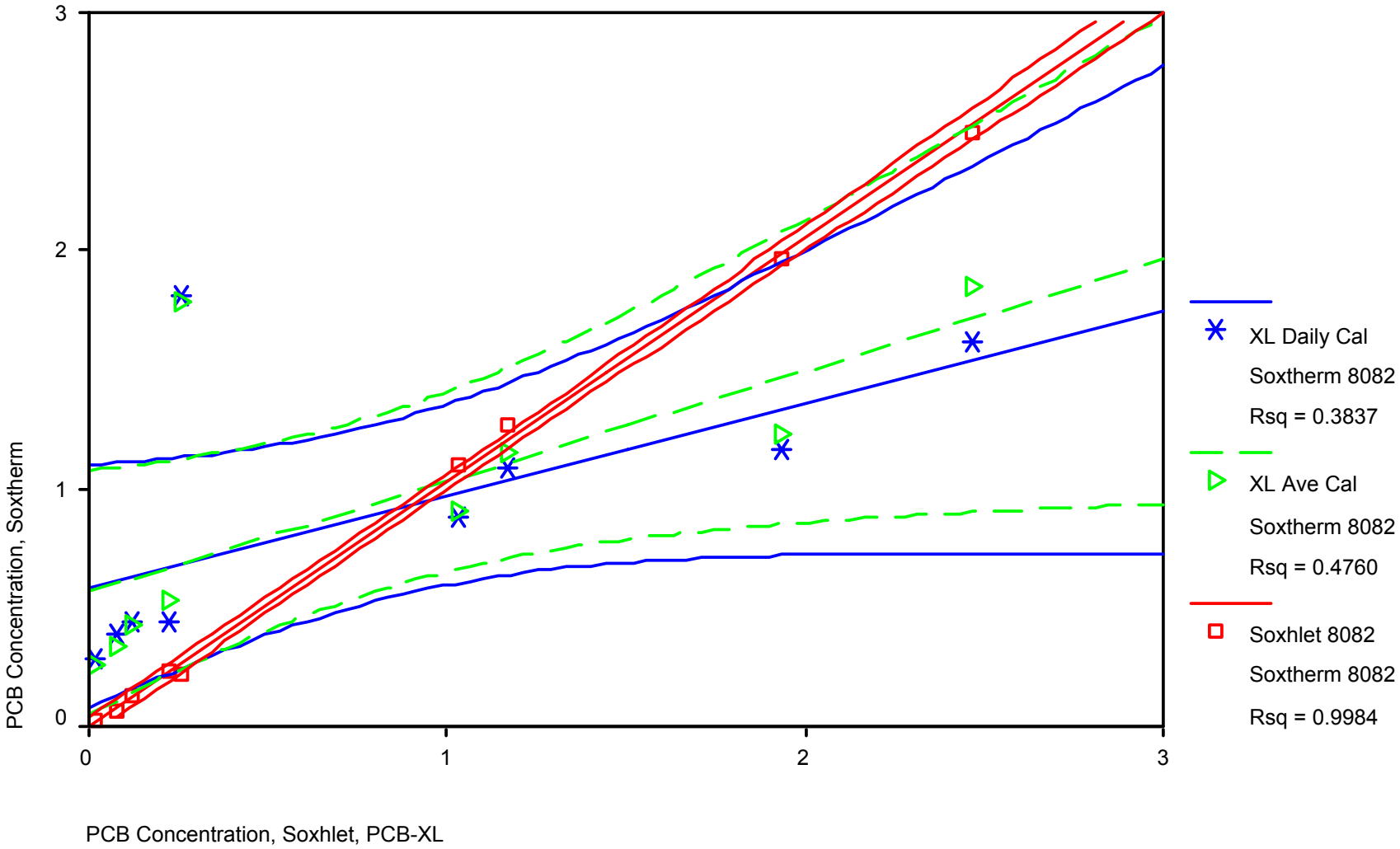

Figure 13
Mean Regression Line with 95 and 99% Confidence Intervals

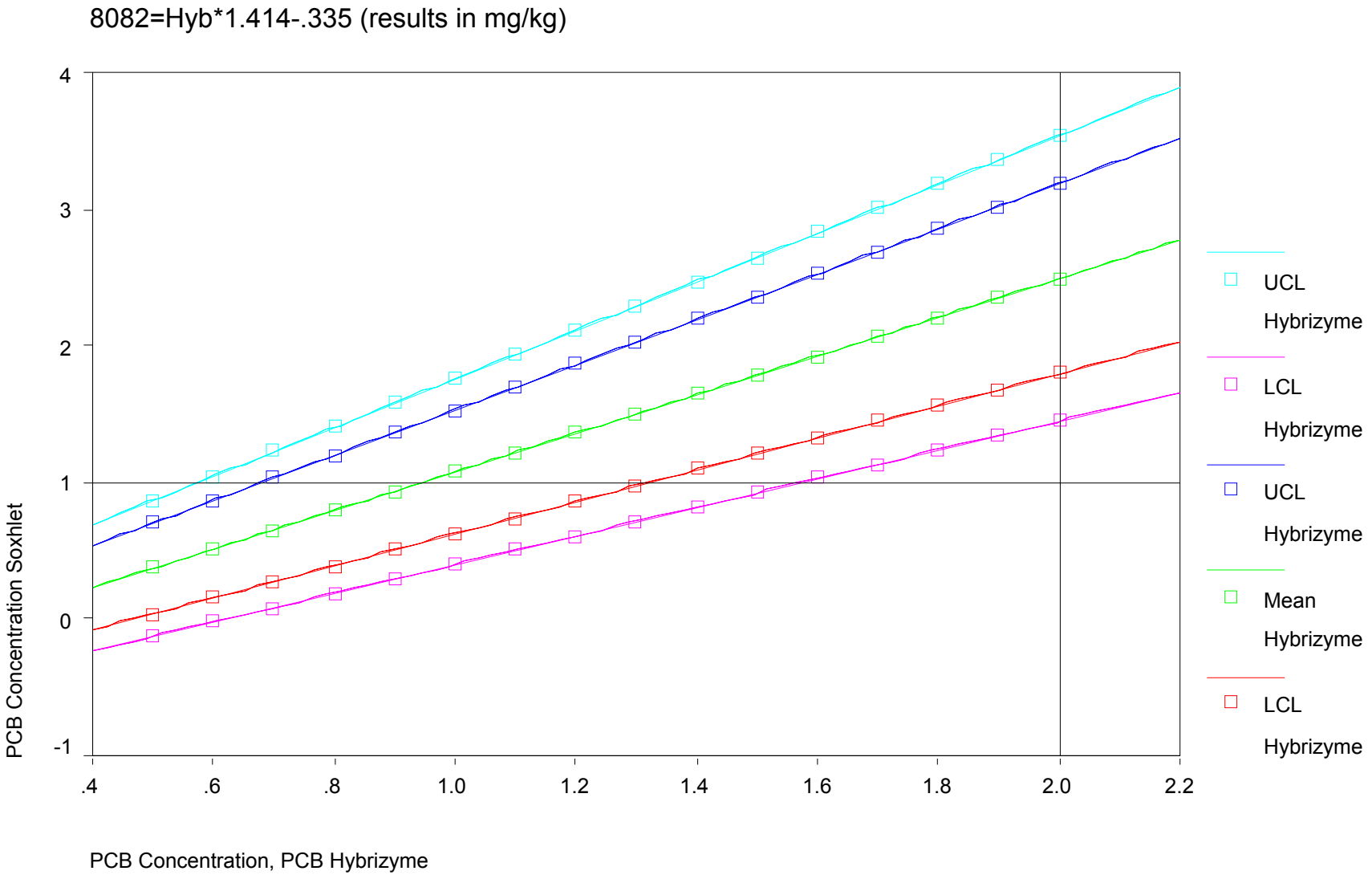

Figure 14
WTM Data Subset >0.3 - <6.0 mg/kg vs. Predicted Hybrizyme Concentration with 99% Confidence Interval